THE EFFECTS OF ATOMIC OXYGEN ON THE OUTGASSING PROPERTIES OF
SPACECRAFT MATERIALS

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ABSTRACT

The Effects of Atomic Oxygen on the Outgassing Properties of Spacecraft Materials

Eli Z. Gurnee

The space environment contains many harsh characteristics that are harmful to spacecraft and threaten the success of space missions. Atomic oxygen (AO) and outgassing are among the chief concerns that spacecraft engineers must design for in order to ensure the safety of a spacecraft. AO is monatomic oxygen (O_1) that is created when Ultraviolet (UV) radiation photochemically disassociates diatomic oxygen (O_2) in space. AO is the dominant atmospheric constituent between 175 and 600 km, and is a great concern in low earth orbits. Orbital AO has an average impact energy of 4.5 ± 1 eV with orbiting spacecraft and is also very reactive; this makes AO very corrosive to spacecraft materials. Outgassing is the process by which trapped and adsorbed gases are expelled from materials. The high temperatures and low pressure of the spacecraft environment exacerbate the process of outgassing. Outgassing is problematic for spacecraft because outgassed material can condense on sensitive surfaces such as optical and thermal surfaces, or the material can create clouds that impede sensors ability to observe their target. While it has been shown that many aspects of the spacecraft environment act synergistically together to further degrade spacecraft performance, there is very little information and data available on the interactions between AO and outgassing. Cal Poly's Space Environments Lab is equipped with an AO simulation vacuum chamber (MAX) and an outgas testing chamber (Micro-VCM) which is capable of testing materials for total mass loss (TML) and collected volatile condensable mass (CVCM) outgassing values. MAX and Micro-VCM were used in tandem to test different spacecraft materials in order to determine if AO exposure had any effect on the respective materials TML and CVCM values. Prior to conducting testing, Micro-VCM was refurbished and validated since it was recently donated to Cal Poly and was not in working order upon arrival. Three Sheldahl materials were tested: aluminum coated 1.0 mil Kapton tape, 2.0 mil Kapton film coated with ITO on one side and aluminum on the other, and 2.0 Kapton film coated with aluminum. The materials were exposed to an average AO fluence of $1.33 \pm 0.130 \times 10^{21}$ atoms/cm² for AO testing. The TML and CVCM results from four of the six tests did not show any significant changes between AO samples and control samples, partially due to large error bars that stem from using a semi-microbalance instead of a full microbalance. However, the AO exposed ITO-Kapton-Al did show an increase in TML from $-0.03 \pm 0.09\%$ to $0.19 \pm 0.08\%$ for one procedure, while the aluminum Kapton tape CVCM decreased from $0.81 \pm 0.12\%$ to $0.63 \pm 0.12\%$ for another procedure. These results show that two materials exhibited a change in their outgassing properties after AO exposure. More testing on the subject is warranted and should be conducted in order to collect more data points and begin defining trend lines that can further describe the effects of AO on outgassing.

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1. INTRODUCTION

1.1 THESIS STATEMENT

There are many aspects to the space environment that degrade spacecraft materials. Engineers must understand the space environment, and spacecraft materials must be tested on the ground to determine their ability to withstand their harsh environment. As such, there are two purposes of this thesis. One purpose is to establish that Cal Poly's newly acquired Micro-VCM machine is fully functional and capable of conducting outgassing tests according to the ASTM E595 outgassing standard. The other purpose of the thesis is to explore whether material exposure to atomic oxygen (AO) will have any effect on the material's outgassing properties, i.e. percentage total mass loss (TML%) and percentage collected volatile condensable mass (CVCM%). Three different materials will be tested and two different procedures will be implemented. Samples will be placed in Cal Poly's atomic oxygen chamber and exposed to AO for 24 hours and then be tested in the Micro-VCM chamber for TML and CVCM. Samples exposed to AO will be compared to control samples that were not exposed to AO. The goal of the thesis is to discern whether samples that were exposed to AO show any increase or decrease in TML and CVCM when compared to the control samples.

1.2 SPACECRAFT ENVIRONMENTS BACKGROUND

A spacecraft system is often broken up and thought of as the main subsystems that contribute to the spacecraft, i.e. attitude determination and control, power generation, propulsion, thermal control, etc. However, the effectiveness and survivability of these subsystems and their

components will be drastically decreased if the spacecraft is not designed to withstand the harshness of the spacecraft environment. In 2003, it was determined that approximately 25% of spacecraft failures to date were a result of interactions with the spacecraft environment.^[31] The spacecraft environment includes the ambient environment that the spacecraft is subjected to (typically a function of orbit) as well as the spacecraft induced environment (the environment generated by the spacecraft's presence).^[16] The spacecraft environment is often grouped into five categories: the vacuum environment, the neutral environment, the plasma environment, the radiation environment, and the particulate environment.^[31]

The vacuum environment refers to the extreme decrease in pressure that spacecraft are subjected to in orbit. The pressure at 100 km above the Earth's surface is over six orders of magnitude below the pressure at sea level; at typical orbital altitudes for objects like the ISS (~350 km) the pressure is over ten orders of magnitude less than on the surface. The vacuum environment can result in high-pressure differentials onboard a spacecraft. Spacecraft must be equipped with vents to allow gases to escape during the ascent to orbit. Missions involving human space travel require pressurized spacecraft; the structural analysis conducted on the spacecraft must ensure that the spacecraft can handle the high-pressure differential. The vacuum environment also contributes to the radiation environment. While Earth's atmosphere filters out much of the UV radiation from the sun, the vacuum environment does not; this means that spacecraft are subjected to higher amounts of UV radiation, and to a wider spectrum of wavelength. The final concern that stems from the vacuum environment is contamination. The majority of spacecraft materials contain trapped gases from the manufacturing process. These gases are under pressure, and tend to bubble out of the material in a phenomenon known as outgassing. The vacuum of space exacerbates this process because there is a very high-pressure

differential between the trapped gases and the ambient environment. Molecules that outgas from a material can collect on surfaces and contaminate them. Outgassing can cause problems for thermal surfaces and optical sensors and degrade their properties.^[31]

While space is often thought of as a perfect vacuum, this is not the case; the Earth's atmosphere extends into space and is prevalent throughout the lower part of the LEO orbital regime. LEO is typically defined as orbital altitudes less than 2000 km, while the neutral environment can be given an upper bound of ~1000 km. The particles that make up the extension of the atmosphere are referred to as neutral particles because they are electrically neutral.^[31] Neutral particles themselves do not contain large amounts of energy, but the high orbital velocities of LEO spacecraft result in detrimental effects on spacecraft. When these particles impact the spacecraft, momentum is transferred between the particles and the spacecraft, which results in aerodynamic drag.^[26] The drag force in LEO is strong enough to deorbit spacecraft; this requires spacecraft to carry enough onboard fuel to perform propulsive maneuvers in order to boost them back into the desired orbital slot. Interactions with neutral particles can also result in a net torque on the spacecraft; reaction wheels or other onboard attitude control devices must be sized to accommodate this torque. Interactions with neutrals can also result in material degradation. Atomic oxygen is a dominant substance in the neutral environment that has the ability to break chemical bonds and oxidize surface materials, resulting in material erosion. Mechanical degradation can occur as well; sputtering occurs when neutrals collide with the spacecraft with enough energy to remove surface molecules. Neutral molecules can also condense on material surfaces of a spacecraft; this can result in the degradation of thermal properties, solar array efficiencies, and optical sensors.^[12]

Plasma is present across all orbital regimes. Plasma is formed when an electron is hit with enough energy to escape the nucleus that it is electrically attracted to. This results in negatively charged electrons and positively charged atoms, or ions. Essentially, plasma forms when electrons have enough kinetic energy to remain free from ions. In LEO, UV radiation has enough energy to ionize the oxygen and nitrogen atoms that constitute the environment; the energy of UV radiation is enough to allow the electrons to escape. This process is called photo-ionization. In higher orbits such as GEO, plasma is formed by photo-ionization as well as transport from other regions. Plasma density is greatest in LEO and decreases as altitude increases, but the GEO plasma is higher energy.^[31] The major effect that plasma has on spacecraft is arcing. Arcing occurs when charges build up on surfaces (arcing is not specific to spacecraft). When two surfaces are charged to a different voltage, the difference in potential causes the charges to redistribute via an electric arc. The ions and negatively charged electrons of plasma can charge surfaces on a spacecraft to allow arcing to take place. There are several modes of arcing, including electrostatic discharge, dielectric breakdown, metallization melt, bulk breakdown, surface breakdown, and induced currents.^[12] Arcing can severely damage electronics onboard a spacecraft by inducing unwanted current through the components.^[16] Other than arcing, plasma is also a concern for spacecraft because it can impede communications, distort navigation data, damage surface materials, and create plasma wakes that affect the performance of nearby spacecraft.^[26]

Any highly energetic particle or photon can be considered radiation. Examples of radiative particles are energetic electrons, protons, neutrons, and ions, while examples of photons include gamma rays and X rays. In space, there are three natural sources of radiation: trapped radiation belts, galactic cosmic rays, and solar particle events. The trapped belts of radiation are

referred to as the Van Allen belts. The Van Allen belts consist of mostly trapped electrons and protons that are trapped in orbit along the Earth's magnetic field lines. These particles are much more energetic than the particles that make up the plasma environment; particles in the Van Allen belts have energy levels on the order of MeV. Galactic cosmic rays are mostly energetic nuclei and originate from interplanetary sources, presumably supernova explosions or particles that have been accelerated by interstellar fields, or the destruction of Alderaan; galactic cosmic rays are higher in energy than the Van Allen belts or solar particle events. Solar particle events are mostly energetic protons that are emitted from the sun during coronal mass ejections.^[31] Radiation can penetrate deep into materials and easily go through outer surfaces of components. For electrical components, this can lead to problems such as connection burnouts, memory errors caused by bit flips, an increase in signal noise, clock resets, and solar cell semiconductor damage. As previously mentioned, UV radiation from the sun degrades spacecraft materials. UV radiation has the energy to break chemical bonds and alter surface properties, such as the absorptance and emissivity of thermal surfaces. Solar radiation also includes radio frequency electromagnetic waves, which can hinder spacecraft communications systems.

The particulate environment refers to the micrometeoroids and orbital debris that exist in space. Micrometeoroids are naturally occurring particles that come from the breakup of comets asteroids. Orbital debris, however, is human made. Orbital debris is clouds of orbiting particles that are left over from human space missions; sources of debris are nonoperational spacecraft, boost stages, solid rocket fuel particles, clamps, de-spin cables, metallic fragments, paint, and remnants from launch vehicle and spacecraft explosions.^{[26][31]} Orbital debris can be divided into three categories based on size. Particles less than 1 cm in diameter have large values of kinetic energy, but they can be shielded against. Particles greater than 10 cm in diameter can be tracked;

this means evasive maneuvers can be implemented to avoid these large particles if necessary. The most problematic particles lie in the middle at 1 – 10 cm in diameter. These particles cannot be tracked, and have velocities ranging anywhere from 7 – 70 km/s. Collisions with particulates can be catastrophic; particulates with such high kinetic energies can damage and destroy spacecraft surfaces and components.^[12] Collisions are also a huge concern for any mission involving sending humans to space; collisions that create holes in spacesuits, shuttles, or capsules can subject humans to a depressurized environment and are very dangerous.^[31]

While it is clear that each specific environment poses a plethora of threats to spacecraft exposed to them, it is not enough to analyze the effects of each environment separately. This is because the separate, destructive, mechanisms found in each environment can actually interact with each other in a synergistic effect that compounds the degradation seen by a spacecraft. For instance, the particulate environment can interact with the plasma and neutral environments when micrometeoroid collisions expose underlying materials; this exposure leaves the materials vulnerable to plasma charging or erosion by neutral atoms. Plasma charging changes the amount of energy required to react with spacecraft surfaces, which can result in greater erosion rates when a charged surface is bombarded by neutral particles such as atomic oxygen. Radiation has been known to increase the amount of outgassed material from the normal outgassing levels, which means spacecraft can be subjected to more contaminants than expected due to radiation exposure. Radiation can also polymerize outgassed material when it collects on surfaces, which darkens the material and causes further surface degradation. Radiation also works synergistically with AO. Incident radiation has enough energy to break chemical bonds, which facilitates AO to react with free surface molecules; this increases the erosion rates of materials.^{[12][27]}

It is clear that understanding the spacecraft environment and its effects on spacecraft is crucial to spacecraft mission designers. In-flight testing operations such as the Long Duration Exposure Facility (LDEF), and the Polymer Erosion and Contamination Experiment (PEACE), which was part of the Materials International Space Station Experiment (MISSE), have contributed enormous amounts of data and information to understanding the spacecraft environment.^{[3][8]} However, orbital tests are expensive and time consuming; these tests are not conducted frequently enough to rely upon for all space environment testing. Therefore, it is very important to establish ground-testing methods that can accurately predict reactions to the space environment.

1.3 MOTIVATION

There are multiple motivations behind this research. As highlighted above, the space environment is an extremely important consideration for spacecraft engineers. The synergistic effects of the spacecraft environment can be even more detrimental than the direct effects that singular mechanisms have. It has been established that radiation can affect outgassing properties due to its high energy and ability to break bonds and chemically alter materials. The same effect has been shown between radiation and AO interactions. This gives rise to the question: can AO exposure affect the outgassing properties of spacecraft materials? There is very little information publicly available about AO and outgassing interactions. If AO does have an effect on outgassing properties, such as increasing the mass of contaminants outgassed from a material, it is important for spacecraft designers to be aware of it. The testing in this thesis will involve exposing the materials to AO and then outgassing the materials separately. This does not reflect the interaction that would occur in space because spacecraft materials on orbit are exposed to AO while simultaneously outgassing. This means that the results of this thesis cannot be directly

extrapolated to predict what would happen in space. However, determining whether materials outgassing properties change after AO exposure will help to form hypotheses on in-space behavior, thus highlighting the motivation between conducting the separate testing.

Another motivation for this thesis is to get the newly acquired Micro-VCM apparatus up and running and to establish its validity. The vacuum chamber was acquired from JPL in a non-functional state and required a lot of work to return it to nominal operations. Once a functioning working order was restored to the machine, it was important to conduct outgassing testing to establish that the apparatus is capable of performing valid outgassing testing.

A final motivation for this project is to establish the correct procedures to test materials between Cal Poly's atomic oxygen chamber and Micro-VCM apparatus. Cal Poly has been given a unique opportunity in that it now has access to two apparatuses that will allow for atomic oxygen and outgas testing to be conducted, testing that has not been conducted in many places up to this point. It is important to establish methods to test materials for AO and outgassing effects in a manner that will yield viable results.

2. BACKGROUND

2.1 OUTGASSING

Outgassing is the process in which a material releases trapped gases over time.^[31] The vast majority of materials, spacecraft related or not, outgas gaseous products throughout their lifetime on Earth; however, the vacuum environment of space exacerbates the outgassing process. Molecules that outgas from a material include water vapor and other species that the material can adsorb, as well as volatile chemicals such as solvents, catalysts, and incompletely polymerized polymers; many volatile chemicals are leftover from improper catalyst/resin ratios or improper curing. There are three mechanisms by which materials can outgas volatiles to the surrounding environment: desorption, diffusion, and decomposition.^{[15][31]}

Desorption is the release of molecules that reside on the surface of a material. Initially, all materials have layers of gases physically or chemically adsorbed onto their surfaces.^{[21][16]} These molecules are held to the surface by physical or chemical forces that have to be overcome to allow the molecules to be released. Diffusion is the process in which gases trapped within the material diffuse to the material's surface. If they have enough thermal energy upon arrival to the surface, the molecules can escape the surface forces keeping them there (essentially undergoing desorption)^[26] and evaporate into the ambient environment. Finally, decomposition takes place when chemical reactions cause complex compounds to divide into smaller substances, or when sublimation of the material takes place. Essentially it is a change in the material, which causes the material itself to outgas. Typically, diffusion accounts for the majority of outgassing, followed by desorption and then decomposition. Desorption contributes to less outgassing than diffusion because there are only so many adsorbed molecules that can be desorbed from the material surface before the layers of gases are all gone. Desorption also takes less time to finish

its outgassing process because adsorbed molecules take less time to be desorbed than trapped gases take to diffuse to the surface and adsorb.^{[16][31][21]}

The outgassing rates and total amount of outgassed material that occurs due to the three described mechanisms depend on a multitude of factors, namely the activation energy, temperature, and processing history of the material in question, as well as the pressure of the material's environment and time that it spends in that environment.^{[31][16]} The activation energy is the energy required for a chemical reaction to take place. In terms of outgassing, activation energy can be looked at as the measure of the strength of the surface binding forces that keep molecules attached to the surfaces of materials.^{[26][31]} Temperature, in the sense of outgassing, is a measure of the thermal energy available for the molecules to undergo reactions. For instance, diffusion requires thermal energy to move gases to the outside of the material. Desorption requires thermal energy to break molecules away from the surface, and decomposition requires energy for the reactions to take place and break molecules of the material down. Therefore, outgassing rates are very dependent on temperature.^[31] The processing of the material comes into play because the amount of volatile gases a material contains depends on what ambient gases it was exposed to during manufacture and processing; a material's treatment is responsible for its adsorption and absorption of gases which with then outgas through diffusion and desorption.^[16] Outgassing rates and total mass loss is also a function of time; it has been observed that metals outgas according to a time dependence t^{-1} and glasses to a dependence of $t^{1/2}$. The three mechanisms of outgassing also have different time dependencies, as well as different activation energies. Typical values of activation energy and time dependency are shown in Table 1.^[31] The table shows that desorption and diffusion have similar levels of activation energy, while decomposition takes a lot more energy to activate. This gives further evidence that

desorption and diffusion contribute more to outgassing than decomposition since it takes much less energy to instigate them as outgassing mechanisms.

Table 1. Activation Energies and Time Dependencies for Three Mechanisms of Outgassing^[31]

Outgassing Mechanism	Activation Energy (kcal/mole)	Time Dependence
Desorption	1 – 10	$t^{-1} - t^{-2}$
Diffusion	5 – 15	$t^{-1/2}$
Decomposition	20 – 80	N/A

Relationships have been formed that can predict the amount of outgassing that a material will experience. These equations serve to show how a few of the factors that affect outgassing totals and rates interact with each other. The rate of outgassing due to diffusion for a given material is given by^[31]

$$\frac{dm}{dt} = \frac{q_o e^{-E_a/RT}}{\sqrt{t}} \quad (1)$$

where dm is the change in mass, dt is the change in time, q_o is the reaction constant which must be experimentally determined for every material and depends on material properties as well as material processing history, E_a is the activation energy, R is the universal gas constant, T is the temperature, and t is the time. The integration of Eq. 1 yields the total mass lost by the material over a given time, as shown by^[31]

$$\Delta m = 2q_o e^{-E_a/RT} (t_2^{1/2} - t_1^{1/2}) \quad (2)$$

where Δm is the total change in mass. While the usefulness of Equation 1 and Eq. 2 depends on the ability to obtain q_o values for materials, the equations serve to show the relationship that activation energy and temperature have with mass loss. The higher the temperature, the higher the mass loss; conversely, the higher the activation energy, the lower the mass loss. The reaction constant is also a very important entity for determining the outgassing that a material will undergo.

The reason outgassing is a problem is that outgassed molecules can collect on surfaces and alter material properties. For this reason, it is important to understand not only how and why materials outgas, but also how outgassed material is transported and deposited onto surfaces. The amount of outgassed material that reaches a surface depends on the outgassing rate of the source and the view factor between the surface and the source.^[31] The view factor for outgassing is a geometric calculation that is similar to the view factor used for radiative heat transfer problems, and is given by the integral^[31]

$$VF = \int \frac{\cos \theta \cos \phi}{\pi r^2} dA \quad (3)$$

where VF is the view factor, θ is the angle between the normal to the outgassing source and the radius vector to the collection point, ϕ is the angle between the normal to the collection point and the radius vector from the collection point, r is the distance between the source and collector, and the integral is performed over the area of the outgassing source, A . Once the view factor has been calculated the arrival rate of mass at a given point can be calculated from the equation^[31]

$$\phi = \sum_s VF_s \frac{dm_s}{dt} \frac{1}{\rho_s} \quad (4)$$

where ϕ is the arrival rate of mass, dm/dt is the outgassing rate from Eq. 1, ρ is the density of the contaminant. The summation is conducted over all possible outgassing sources, denoted by the subscript s , since there can be multiple sources outgassing to a point at one time. It is important to note that even if an outgassing source doesn't have a direct view factor to a surface, contaminants from that source can still affect the surface. Outgassed material can be deposited on one surface, and then be reemitted and deposited on another surface that is out of view of the original source; this interaction was shown on space shuttle missions, when contaminants were able to reach sensitive payload sections of the shuttle bay even without direct lines of sight to them.^[16]

Just as not all of the outgassed material from a source will reach a collector point, not all the material that reaches the point will collect on the receiver surface. In order to collect on a surface, outgassed material must adhere to the surface and reach thermal equilibrium; surfaces made from different materials have different surface capture coefficients, a coefficient that describes the probability that an incident molecule will become thermally accommodated upon collision with a surface.^{[21][31]} This means that the probability of outgassed material condensing on a surface is directly related to the temperature of the surface. Once outgassed molecules collect on a surface, they will stay there until they acquire enough energy to escape the binding forces of the surface. The average time for a contaminant to reside on the surface can be calculated with the equation^[31]

$$\tau = \tau_o e^{E_a/RT} \quad (5)$$

where τ is the residence time, and τ_o is a typical oscillation period of the molecule on the surface and is $\sim 10^{-13}$ seconds. An example of how big an effect temperature has on contaminant selection

is water vapor; water vapor has a residence time of 10^{24} seconds on a 100 K surface, but only 0.25 seconds on a 300 K surface.^[31] Clearly, outgassed material is more likely to condense on a surface if the surface is cold, which is especially problematic for sensors and optical surfaces that are kept at cool temperature.

Deposited material can affect and degrade thermal surfaces, solar arrays, instrument optics, sensors, and other systems.^[10] Thermal surfaces are chosen for their absorptance and emissivity properties; often it is ideal to have low absorptance and emissivity for outer layers of MLI, or low absorptance and high emissivity for radiators. Outgassed contaminants that collect on thermal surfaces increase the absorptance and emissivity of the material. Contaminants have three modes in which they can degrade thermal properties: they can completely block the surface, they can trap radiation, and they can become sites that scatter radiation.^[10] It is important for engineers to take thermal property degradation into account when designing a spacecraft's thermal control system; the spacecraft must be able to maintain satisfactory temperatures at the beginning of life as well as at the end of life when properties are degraded.^[10] Similarly to thermal surfaces, optical surfaces and sensors can be degraded when contaminants build up on the surface and form blockages. However, optics and sensors face another problem as well; outgassed contaminants that do not end up collecting on a surface can find their way in between a detector and the object it was designed to observe. When this happens, the field of view of the detector is obstructed by the contaminants and the signal being received by the detector will be diminished. Star trackers have been shown to suffer degraded tracking ability due to contaminant clouds on orbit.^[6] Outgassing effects are not limited to surface property degradation, as composite materials have been shown to contract and undergo dimension changes and deformation due to the outgassing of gases such as water vapor.^{[29][6]}

There are a number of ways to mitigate the effects of outgassing. First, it is important to sensor apertures in a way to minimize contamination; care should be taken so that outgassing sources do not have a direct view factor to optical surfaces and sensors. Similarly, outgassing sources can be arranged to vent volatiles away from sensitive spacecraft surfaces. Covers can also be implemented to disallow outgassed material to reach surfaces. It is very important, and is a common practice, to bake out spacecraft components and materials before launch. This bake out is meant to induce outgassing from materials so that a majority of volatiles will already be outgassed before the spacecraft reaches orbit.

It is also very important to be able to test materials on the ground so that engineers know how much to expect specific materials to outgas. ASTM E595 was created for this purpose to test for Total Mass Loss (TML) and Collected Volatile Condensable Mass (CVCM) for materials. TML is the total mass that is outgassed from a material, while CVCM is the total amount of outgassed substances that collect on a cold surface. Both metrics are calculated as percentages of the original material mass. In order to test for these values, materials are placed in a vacuum of less than 5×10^{-5} Torr for 24 hours. The materials are heated to 125°C to induce outgassing, and are placed in front of collector plates that are kept at 25°C. Remembering Eq. 2 and Eq. 5, this makes sense conceptually since a higher material temperature is conducive to outgassing because it provides thermal energy; similarly, the lower the temperature of a surface, the more likely it is that volatile chemicals will condense on the surface, thus the 25°C temperature of the collector plate. The material sample and collector plate are weighed after the 24 hour test to determine TML and CVCM. Typically, a material is considered “space rated” if it exhibits a TML of <1% and a CVCM of less than 0.1%, so ASTM E595 allows engineers to test materials and know whether they are fit for space missions or not.^[1] ASTM E595 will be

discussed in more depth in future sections as the testing conducted for this thesis followed the procedures laid out by the standard.

As mentioned in the introduction, there are many aspects of the spacecraft environment that act synergistically; outgassing is no exception. UV radiation has been known to exacerbate the effects of outgassing in multiple ways. Incident UV radiation on a surface has been shown to affect the deposition of outgassed material. The presence of UV radiation during the outgassing process can accelerate deposition rates onto surfaces; also, UV radiation can cause contaminant deposition on a surface even when deposition would not normally occur on that surface. Not only does UV radiation affect the deposition of contaminants, but it can also affect the contaminants that have already been deposited on the surface. UV radiation can photopolymerize the deposition on a surface; this process darkens the film of contaminants and makes it more disruptive to surface properties.^[21] The effects of UV radiation are not limited to contaminant deposition; it has also been shown to increase outgas rates for materials. Recent studies have shown that the photodecomposition effect of UV radiation increases the desorption and decomposition outgassing mechanisms of materials.^{[17][18]} Atomic oxygen has also been shown to interact with outgassed contaminants; a deposited contaminant layer on a quartz substrate was exposed to AO and resulted in a stable SiO₂ contamination layer that decreased the optical transmittance of the material.^[13]

As UV radiation has been shown to affect the outgassing rates of materials, it is natural to wonder if atomic oxygen exposure can have the same effect. UV radiation increases outgassing rates through decomposing surface layers of material, and disassociating molecules into molecules that are more easily desorbed from the surface. AO induces the same types of effects

onto the surfaces of materials, so it is possible that AO exposure could affect the outgassing rates of materials.

2.2 ATOMIC OXYGEN

Oxygen found within the lower levels of Earth's atmosphere is molecular diatomic oxygen, or O_2 , meaning that oxygen atoms are always found in pairs. This is because diatomic oxygen is stable and has a large disassociation energy of 5.12 eV. The disassociation energy is the energy required to break the O_2 diatomic bond and create O_1 ; there is nothing naturally present within the lower levels of the Earth's atmosphere (parts of the Mesosphere and below) with that amount of energy.^{[10][26]} At altitudes high above the Earth, however, UV radiation with energy levels exceeding 5.12 eV is present. Earth's atmosphere absorbs UV radiation of all spectrums, so some of the UV radiation from the sun does not reach the Earth's surface. Short-wavelength (< 243 nm)^[10] UV radiation from the sun is completely absorbed by the atmosphere so none of it ever reaches the Earth's surface. UV radiation of this wavelength has energy levels exceeding the 5.12 eV disassociation energy of O_2 . Oxygen is still abundant at altitudes high above the Earth, and the short-wavelength UV radiation is prevalent at these altitudes because it has not been completely absorbed into the atmosphere yet. Therefore, there is a region between the altitudes of 60 and 800 km above the Earth where high energy UV radiation photochemically disassociates molecular diatomic oxygen into singular atomic oxygen, or AO.^[26] Not only are the conditions at these altitudes perfect for breaking oxygen bonds, but the mean free path in this environment is long enough ($\sim 10^8$ m) that there is a very low probability that the AO atoms will re-bond together as O_2 or form Ozone (O_3).^[10] This region corresponds to the orbital regime known as Low Earth Orbit (LEO); AO effects are very important to consider for LEO spacecraft.

While AO is abundant between 60 and 800 km, it is actually the dominant atmospheric constituent between the altitudes of 175 and 600 km.^[26] This is illustrated in Figure 1, which shows the number density of different atmospheric constituents relative to altitude (the reference does not specify whether the data shown in Figure 1 is for solar min, solar max, or average solar flux). Figure 1 shows that while AO is the most abundant constituent between 175 and 600 km, the number density of AO begins to decrease dramatically as the altitude reaches 700 km and higher. This means that while it is very important to consider the effects of AO throughout the LEO regime, AO effects become less consequential as altitudes approach 800 km and higher; at higher orbits, AO effects are negligible and do not need to be considered.^[12]

Figure 1. Number density of atmospheric constituents relative to altitude based on the NRLMSISE model.^[12]

AO density is not only a function of altitude. The solar cycle causes values of AO density to vary throughout the LEO regime, making it difficult to accurately predict AO flux and fluence for a specific altitude for specific times.^[4] The AO flux is a measure of the rate of AO particles passing through a given area; the flux has units of atoms/cm²/s. The AO fluence is the total number of particle collisions over a certain amount of time, or simply the AO flux multiplied by the time in question. The units of fluence are atoms/cm². The 11-year solar cycle has a large effect on annual AO flux and fluences; the AO fluence can fluctuate from less than $1.0 \cdot 10^{21}$ atoms/cm² at solar minimum to over $3.0 \cdot 10^{21}$ atoms/cm² at solar maximum. Figure 2 compares the difference in AO fluence each year over the 11-year solar cycle, starting in 2004. The data shown in this figure was calculated using the MSIS-86 atmospheric model, meaning the figure shows projections, not actual values.

Figure 2. AO fluence (in ram direction) over the 11-year solar cycle.^[10]

Atomic oxygen is important to understand because it is erosive to spacecraft materials. AO's erosive tendencies stem from the high impact energy of the AO atoms with spacecraft surfaces, as well as from the highly reactive nature of oxygen.^[16] Oxygen atoms have six electrons in their valence shell, meaning they are ready to react with other elements. Oxygen atoms are ready to oxidize (take electrons from) other atoms in order to obtain a full valence shell of eight electrons. For instance, oxygen will readily react with carbon and accept two electrons from the carbon atom. The impact energy of AO on spacecraft is high enough to break chemical bonds of the atoms that reside on the surfaces. Once the bonds have been broken, other AO atoms are able to bond with (oxidize) the now free surface atoms.^[16]

While the reactivity of oxygen is straightforward, the impact energy of AO is more complicated as it depends on multiple things. The ambient AO energy is based upon the thermal energy of the thermosphere or exosphere, depending on where in LEO the spacecraft is.^[12] The total impact energy of AO, however, includes kinetic energy from the orbital speed of the spacecraft as well as the Earth's atmosphere co-rotation velocity and the thermal velocity of the atoms themselves. The total energy also depends on other orbital parameters, such as orbital inclination.^[10] This makes it difficult to assign an energy value to AO. However, in Reference 10 Banks et al established that for a spacecraft in a 400 km, circular, 28.5° inclined orbit, the average impact energy of AO is $4.5 \text{ eV} \pm 1 \text{ eV}$. They go on to note that the impact energies decrease as altitude increases, which makes sense because the spacecraft orbital speed is decreasing. Making simplified calculations for the AO ambient energy and kinetic energy can help to confirm the value of 4.5 eV. Assuming an average thermosphere temperature of 1000 K^[10], and utilizing Boltzmann's constant of $8.617 \times 10^{-5} \text{ eV/K}$ to convert to eV, the ambient

energy of AO is only about .08 eV. However, taking the orbital speed of a 400 km altitude spacecraft into account (7.67 km/s) and plugging it into the kinetic energy equation,

$$E = \frac{1}{2}mv^2 \quad (6)$$

where E is energy in Joules, m is mass and is equal to $2.65686227 \times 10^{-26}$ (16 amu for one AO atom), and v is velocity in km/s, yields an energy of 7.82×10^{-19} Joules. Converting Joules to eV yields a total impact energy of 4.96 eV for AO at 400 km, which is well within range given by Reference 10. It is important to note that this is just an average impact energy, meaning that some AO atoms will be colliding with the spacecraft at even higher energies. It is also important to note that AO properties can be described with a Maxwell-Boltzmann distribution.^[12] The high velocity tail of the Maxwell-Boltzmann actually results in some AO atoms catching up with surfaces of the spacecraft other than the ram-facing surface. This means that there are small values of AO flux incident on surfaces 90 degrees off of the ram surface, and even on the anti-ram surface of the spacecraft. These fluxes are small percentage points of the ram-facing flux (Reference 10 showed surfaces with normals 90 degrees off of the ram-facing direction received fluxes at 4% of the value of the ram-facing surface), but they are still enough to interact with and erode materials.

The process of AO erosion can be illustrated by looking at one possible mechanism of surface erosion for a graphite surface subjected to AO. Again, this is only one possible mechanism of AO erosion but it serves as a good example. The process is shown in Figure 3. The disassociation energy of a carbon-carbon bond is 7.4 eV. While it was established that AO has an average collision energy of 4.5 eV, some AO atoms can impact a surface with energy levels exceeding 7 eV. When an AO atom collides with the graphite surface with an energy of 7

eV, the carbon-carbon bond is broken and there is a finite probability that a surface oxide (carbon monoxide) will form with a bond strength of 13.1 eV. If yet another AO atom then strikes the surface with an energy greater than 1.7 eV, enough energy can be transferred to the surface to allow the carbon monoxide to form a triple bond and escape in the gaseous phase. This leaves a vacancy on the surface where the original carbon was, i.e. the surface was eroded.^[16]

Figure 3. Atomic oxygen erosion process for a graphite surface.^{[12][16]}

AO erosion falls into two categories, erosion caused by the formation of volatile oxides and erosion caused by the formation of oxides that do not adhere to the eroded surface. The erosion of graphite shown in Figure 3 is an example of erosion due to the formation of volatile

oxides, since CO is a volatile oxide. Examples of oxides that do not adhere to their parent surfaces are silver oxides.^[16] Regardless of what mechanism a material experiences erosion through, all materials can have their susceptibility to AO quantified through a constant known as the reaction efficiency (RE). The reaction efficiency is a constant used to compare the outgassing rates of different materials; a higher reaction efficiency means that a material will react more with AO and will experience more mass loss.^[12] The reaction efficiency is measured in volumetric material loss per incident AO atom, cm³/atom. It can be used in the equation,^[31]

$$dm = \rho_t RE \phi dA dt \quad (7)$$

where dm is loss of mass in grams, ρ_t is the density of the target material, ϕ is the AO flux in atoms/cm²/s, dA is the surface area in cm², and dt is the time of exposure in seconds, in order to predict the mass loss a material will experience when subjected to AO. Reaction efficiency can be a function of surface temperature, AO flux, coincident UV radiation, surface contamination, and other variables. However, through on orbit experiments such as LDEF and MISSE PEACE, and testing conducted on the ground, reaction efficiencies have been determined for multitudes of materials.

Table 2 shows reaction efficiencies for a few of the most popular materials used on spacecraft.

Atomic oxygen reacts with polymers, carbons, and metals; AO has the ability to form oxygen bonds and erode the surfaces of all of them.^[10] Polymers, specifically hydrocarbon polymers, typically have the highest reaction efficiencies when exposed to AO.^[12] It is very important to understand the effects that AO erosion can have on these polymers since they are usually chosen to implement on spacecraft because of their desirable material properties, including thermal properties. Numerous erosion mechanisms take place on unprotected

polymers. When exposed to AO, many polymers experience hydrogen abstraction, oxygen addition, or oxygen insertion. These interactions lead to volatile oxidation as well as the erosion of hydrocarbon materials.^[10]

Table 2. Reaction Efficiencies for standard spacecraft materials.^[16]

Material	RE ($\text{m}^3/\text{atom} \times 10^{-30}$)
Kapton	3
Mylar	3.4
Tedlar	3.2
Polyethylene	3.7
Teflon FEP and FE	<0.1
Carbon	1.2
Polystyrene	1.7
Polymide	3.3
Platinum	0
Copper	0.05

Pits and cones, which are caused by the bombardment of energetic AO atoms on the ram-facing surface, form on the surfaces of polymers which results in rough texturing of the surface and material thinning, which is shown in Figure 4. The change in texture increases the diffuse reflectance of the material and decreases the specular transmittance; these changes also result in changes to the thermal emittance and absorptivity of the material.^{[10][12]} Since the materials were most likely chosen for their thermal properties to begin with, it is extremely important to understand and design for the thermal property changes that materials undergo when expose to

AO. Engineers must take into account the fact that the absorptivity and emittance of a given material will not be the same at the end of life as it was at the beginning of life.

Figure 4. Scanning electron microscope photograph showing surface roughness (pits and cones) of Chlorotrifluoroethylene after AO exposure.^[10]

The mechanisms of erosion become more complicated when the polymers in question make up multi-layer blankets. MLI is often comprised of multiple different polymers that make up each layer; this means that each layer will have a different reaction efficiency and will respond to AO differently. Also, MLI is susceptible to a phenomena known as undercutting, which takes place when a layer of high reaction efficiency becomes exposed and erodes away the material beneath an existing cover layer.

Thermal properties are not the only thing to worry about when choosing materials and designing LEO space missions. AO can affect structural components as well. While most metals are resistant to AO degradation, organic composites are becoming more common to implement on spacecraft due to their high strength to weight ratio. Composites, however, are susceptible to AO erosion. In 2001, Shin et al in Reference 26 conducted testing to determine the effects that thermal distortion and the LEO environment could have on a composite honeycomb solar array.

They established that the most detrimental environmental effect on all organic matrix composites was material loss due to AO. AO erosion was shown to inflict dimensional changes, mass loss, and degradation of mechanical properties on the composite solar array. The strength and stiffness of graphite/epoxy composite materials was shown to decrease after exposure to AO; this is an important effect to be able to predict or mitigate for structural analysis purposes.^[30]

Silver is one of the few metals that is negatively affected by AO (osmium is another example). Silver is an example of a material that reacts with AO to form oxides that do not adhere to their parent surface. This results in spalling of the silver since silver oxide spalls from the underlying metal, and in turn allows for continuous oxidation of the silver. This is problematic as typical solar cells are connected to each other on a solar array with thin silver wires called solar interconnects. The spalling of silver due to AO exposure has caused silver solar interconnects on LEO spacecraft to wear away completely, resulting in the failure of the solar array.^{[12][10][31]}

It is important to note that AO interaction does not always result in the erosion and degradation of a surface. As mentioned earlier, most metals do not react with AO to negative consequences. When AO oxidizes the surfaces of most metals, nonvolatile metal oxides are produced. These oxides are stable and end up protecting underlying materials from AO erosion; this phenomenon is called fixing since the oxides are stable and become fixed on the surface.^[12]
^{[10][6]} Silicone is an example of a material that undergoes AO fixing as SiO_2 is formed when silicone interacts with AO. While it is advantageous for a material to form a layer of stable oxides that protects underlying materials, there are some drawbacks; the fixed surface oxides that form on the surface of silicones have been known to cause cracking and flaking of the surface.^{[10][12]} An example of this material cracking is shown in Figure 5.^[10]

Figure 5. Electron microscope photograph of DC 93-500 silicone showing cracking after AO exposure.^[10]

Even for materials that do react and erode when exposed to AO, the effects are not always detrimental. For instance, in Reference 21, Panickar et al studied the adhesion problem associated with silicone elastomer compound seals that are used in space.^[24] Silicone elastomers are ideal to create seals out of because of their low leak rate, compression force rating, and outgassing properties. However, one drawback to silicone elastomers is their tendency to adhere to metal surfaces; a reusable seal must be able to readily release from its mounting face, making adhesion an undesirable quality. Panickar et al explored whether atomic oxygen exposure would reduce the adhesion of the silicone elastomer seals. They established that an AO pretreatment with a fluence of 1.3×10^{20} atoms/cm² completely mitigated the seal adhesion problem. This is an interesting way to harness the erosive properties of AO, properties that usually are unfavorable for spacecraft materials. Care must be taken, though, when utilizing AO as a pretreatment for seals; AO has also been shown to increase the leak rate of space grade seals.^[25]

There are multiple mitigation techniques to increase the durability of polymers. The most common way to protect polymers is applying thin film metal or metal oxide to the top surface of the polymer as a surface coating.^[12] Possible coating materials include SiO₂, Al₂O₃, ITO, germanium, silicone, aluminum, or gold.^[10] These coatings form stable oxides that serve to protect the underlying polymer. Only a thin layer (~100 nm) of metal or metal oxide is necessary to guard against AO erosion. Care has to be taken when implementing this method because the protective coatings can introduce new problems of their own, such as the silicone cracking problem that was highlighted above.^[12] Keeping the thickness of the coating thin helps to diminish the likelihood of cracking or spalling.^[10]

Surfaces can also be modified to make them more durable. This technique involves implanting metal atoms into the surface of the polymer or chemically modifying the surface so that the polymer contains silicon atoms in and near its surface.^[10] Alternative polymers can also be used that were altered to contain metal atoms within the polymer that form stable oxide coatings when exposed to AO.^[10] These methods, as well as the simple thin coating method from above, are good for protecting polymers against AO effects, but still involve possible anomalous effects that stem from adding other substances to the polymer. A solution to AO erosion that does not involve altering polymers is to use non-carbon based polymers. An example of such a polymer is siloxane. Siloxane's reaction efficiency is far smaller than typical organic polymers, which diminishes the need for protective coatings.

Atomic oxygen has been proven to act synergistically with UV solar radiation. As mentioned previously, the Earth's atmosphere filters out UV radiation of wavelengths below 290 nm. Radiation in this wavelength range is referred to as vacuum ultraviolet radiation, or VUV. The smaller the wavelength of the radiation is, the more energy it has (on the order of 6 – 12 eV

for VUV). VUV has sufficient energy to break atomic bonds on the surfaces of spacecraft material. When materials are subjected to AO and VUV in tandem, the VUV can break atomic bonds on the surfaces of the materials. When the VUV breaks the bonds, it leaves atoms ready for the AO to react with and oxidize. In this way, AO and VUV act synergistically. Synergistic relationships are common in the spacecraft environment. As UV radiation has been shown to affect outgassing properties, it is pertinent to explore whether the oxidation and degradation of materials due to AO exposure results in a change in material outgassing properties such as TML and CVCML.^[31]

2.3 ATOMIC OXYGEN & OUTGASSING

There is very little information available on whether AO has any synergistic effect on outgassing. It is fair to infer from this lack of information that there has not been much testing conducted to determine if there is any relationship between AO and outgassing. However, there is very good reason to conduct testing since it is very easy to see why AO may have an effect on outgassing.

Studies have shown that UV radiation exposure increases the amount of outgassed contaminants from a material and the outgassing rates of a material.^{[17][18]} While AO interacts with materials similarly to UV radiation in that it has the energy to break bonds chemical bonds, AO has the ability to oxidize the free atoms on the surface and cause erosion. It is possible that any or all of the three mechanisms of outgassing could be affected by AO. The breaking of bonds and oxidation that occurs on material surfaces due to AO could change the amount of material desorbed from the surface. The erosion of material due to AO could allow more or less trapped gases to diffuse to the surface and be outgassed since the eroded layers of the material could be easier or harder for gases to diffuse through. Decomposition is essentially the outgassing of the

degraded material itself. The degradation of material through AO erosion could result in lower activation energies needed to release atoms of the material in the decomposition outgassing process. These mechanisms could be affected in a way that results in materials outgassing either more or less material than is typical, or there could be no relationship between AO and outgassing at all.

One study (Reference 7) did show a relationship between AO and outgassing.^[7] The study used size #2-106 S0383-70 and ELA-SA-401 O-rings to conduct testing. The results of the study are shown in Figure 6. The study tested the O-rings after greasing them as well as baking them out + exposing them to AO, but the results that are pertinent to this thesis are the “As-received” and “with AO” columns shown in Figure 6 for both the S0383-70 and ELA-SA-401 O-rings. Figure 6 shows that AO pretreated S0383-70 O-rings experienced a slight decline in both TML and CVCMM when compared to the “As-received” set, while the ELA-SA-401 O-rings showed no significant difference between the sets that did and did not receive AO pretreatment. It is interesting to note that even though the decrease in outgassing that was experienced by the AO pretreated S0383-70 O-rings was within the error bars, the study still concluded that there was a slight decrease in outgassing due to AO for that material. This is important to highlight because the results of this thesis involve large error bars that would seemingly render some observed differences in outgassing insignificant. However, as the study in Reference 7 shows, differences in outgassing can be so small that even small noticeable trends are still worth discussing.

Figure 6. Results of study from Reference 7.^[7]

3. EXPERIMENTAL APPARATUSES

3.1 MICRO-VCM OUTGASSING CHAMBER

In the summer of 2013, JPL donated its Micro-VCM (Micro Volatile Condensable Mass) vacuum chamber to Cal Poly's spacecraft environments lab. The chamber will be referred to as "Micro-VCM" throughout this thesis. The purpose of the chamber is to test for the TML and CVCM values of spacecraft materials; the chamber was designed based on ASTM E595, which dictates procedures, layout, and dimensions for a TML and CVCM apparatus. Upon donation, the Micro-VCM chamber was not functional and had not been utilized for outgas testing for years. A major component of this thesis was reinstating the Micro-VCM chamber to full functionality and ensuring it was capable of collecting TML and CVCM data; only after the chamber was established as a viable outgas testing apparatus would it be able to be applied to research whether AO has any effect on outgassing properties of materials. This section includes a detailed description of how the apparatus works and accomplishes TML and CVCM testing, as well as a summary of changes and fixes that were made to make the chamber operational.

The entire Micro-VCM apparatus is shown in Figure 7. Although the Micro-VCM chamber is a relatively complex system with many components, its main function is to heat material samples to 125°C and cool collector discs to 25°C. The chamber must have the capability of maintaining these temperatures while achieving a pressure of $\leq 5 \times 10^{-5}$ torr. These temperature and pressure values are outlined in ASTM E595. Conceptually, these temperatures and pressure stem from the ideas discussed in the outgassing background section, specifically Eq. 2 and Eq. 5 which show that temperature affects the total mass outgassed by a material and how long contaminants will stay adhered to a surface. The 125°C temperature provides the thermal energy required for materials to outgas, and the 25°C temperature provides a surface

cold enough to collect volatiles. The pressure of $\leq 5 \times 10^{-5}$ torr is sufficient to facilitate the outgassing process.

Figure 7. Micro-VCM testing chamber.

The vacuum chamber achieves a pressure of $\leq 5 \times 10^{-5}$ torr through the use of a Welch 1374 mechanical vacuum pump and a CTI-Cryogenics Cry-torr 8 cryogenic vacuum pump. The mechanical pump is sufficient to reduce chamber pressure to below 150 mTorr; the cryogenic pump then takes over to achieve pressures lower than 5×10^{-5} torr as outlined by ASTM E595. Pressure between 1 mTorr and 760 torr is measured using a Granville-Phillips 275 Convectron Gauge and read off of a Granville-Phillips 316 Vacuum Gauge Controller. Pressure lower than 1 mTorr is measured with a Granville-Phillips 274 Ion Gauge and read off a Granville-Phillips 330 Ionization Gauge Controller. The test chamber is also equipped with a digital Cryo Connections Model 2 Cryopump Monitor, which is used to monitor the temperature of the cryopump as it is cooling down and running nominally. The cryopump is run off of either a CTI-Cryogenics

1020R helium compressor of CTI-Cryogenics 8200 helium compressor. A full schematic of the Micro-VCM vacuum chamber is shown in Figure 8.

Figure 8. Schematic of the Micro-VCM apparatus.

The actual test apparatus of the Micro-VCM machine is comprised of a sample compartment bar and a cold plate. The cold plate holds copper collector discs, which are used to collect the volatile condensable mass that is expelled from the material samples. The cold plate and discs are shown in Figure 9. Figure 9 shows how the copper collector discs fit onto the cold plate. The figure also shows how the cold plate is cooled. The bending plumbing that lies to the right of the copper discs is welded onto the plate; cool water is run through this plumbing to actively cool the plate down to 25°C. The water is supplied by a Brinkmann MGW Lauda RM 20 water-cooling unit which is able to keep a reservoir of water cooled at a given temperature and

cycle water through the plumbing to whisk heat away from the plate; the water-cooling unit can be seen in Figure 7 sitting to the left of the chamber's bell jar.

Figure 9. Cold plate with copper collector discs.

Figure 9 also shows the sample compartment bar, which is detached from the cold plate in the figure. Figure 10A shows what it looks like with the sample compartment bar closed and bolted to the cold plate. The only conductive connections between the sample compartment bar and the cold plate are three screws, which helps to limit heat transfer between the two entities. Figure 10B shows a straight-on, close up view of the sample compartment bar. This view shows a circular compartment with a through-hole that leads to the cold plate. This circular compartment is where material samples are placed within the bar. The figure also shows the copper cover plates that are used to cover the opening of the compartment once the sample is placed inside. There are a total of nine circular sample compartments on the bar, which means

nine samples can be tested at once. When both sample bars are functioning properly, this means that a total of 18 samples can be tested at once; however, currently only one bar is operating so the system is limited to 9 total samples.

Figure 10. A) Closed view of cold plate and sample compartment bar. B) Straight-on view of heater compartment bar.

The sample compartment bar is heated to raise the temperature of the samples within it to 125°C. In order to accomplish this temperature, a resistor with total resistance of 26 Ω is welded to each sample compartment bar, for a total of two resistors in parallel. Current is run through the resistor, which dissipates heat into the copper bar and warms it up to 125°C. Power is supplied to the resistor by a Superior Electronics Powerstat Variac power supply. This circuit is shown in Temperature is measured with T-type thermocouples; one thermocouple is placed on the top of the compartment bar and one is placed at the bottom to ensure the temperature of the bar stays at

$125 \pm 1^\circ\text{C}$. Similarly, two more thermocouples are placed at the top and middle of the cold plate to ensure it stays at $23 \pm 1^\circ\text{C}$. Temperatures are read off of an Omega HH506 thermometer.

Figure 11. Circuit for heating sample compartment bar. 20 V is the typical setting on the Variac for 125°C .

During a test, a material sample will be heated within the sample compartment in the sample compartment bar. The through-hole that is shown in Figure 10B gives the material sample a straight-on viewfactor to a single copper collector disc. This means that any volatile, condensable material that is expelled from the material during the outgassing process will collect on the collector disc, which is being kept at 25°C via the cold plate. There are barriers in place between collector discs on the cold plate that diminish the likelihood that cross contamination of collector plates will occur. The two values to be gleaned from tests conducted in Micro-VCM (according to ASTM E595) are TML and CVCM. TML can be calculated solely by using the mass of the sample material before and after the test with the equation,

$$TML = \frac{S_I - S_F}{S_I} \times 100\% \quad (8)$$

where S_I is the initial sample mass and S_F is the final sample mass after a 24 hour test, and multiplying by 100 results in TML as a percentage. In order to calculate the CVCM of a material, both the masses of the disc and the material sample must be used in the equation,

$$CVCM = \frac{C_F - C_I}{S_I} \times 100\% \quad (9)$$

where C_F is the final mass of the collector disc and C_I is the initial mass of the collector disc. It is important to note that TML is calculated as a percentage of mass *lost* from a material, while CVCM is calculated as a percentage of mass *gained* by the collector disc. According to ASTM E595, a witness collector disc shall always be placed in front of an empty compartment with the rest of the samples and discs in order to measure whether contamination is occurring. If a witness disc collects a significant amount of mass during a test, it can be used as a correction factor for the CVCM calculation like so:

$$CVCM = \frac{C_F - C_I - G_w}{S_I} \times 100\% \quad (10)$$

where G_w is the mass gain of the witness disc.

It is important to note the many changes and fixes that Micro-VCM underwent before becoming fully capable of collecting TML and CVCM data. For instance, chamber came equipped with a diffusion pump to achieve a pressure of $\leq 5 \times 10^{-5}$ torr; the diffusion pump required access to liquid nitrogen, which the space environment's lab does not have access to. Therefore, a cryopump was purchased and installed to replace the diffusion pump. Micro-VCM came with its own temperature and pressure monitors, but they were very old and were unreliable. The Granville-Phillips pressure gauge controllers mentioned earlier in this section were purchased and installed, and the Omega thermometer was utilized for temperature measurements instead of relying on the stock thermometers. The thermocouples that came with that apparatus were all severed and in certain places bare metal was exposed that allowed for unwanted contact with other metal that could hinder correct temperature measurements; great care was taken to solder the thermocouples back together and ensure all metal was wrapped in heat shrink.

The sample compartment bar was designed to heat up by implementing a circuit that included four resistors in parallel before reaching the resistor welded onto the copper bar. This would have resulted in the inability to change the current flowing through the resistor on the bar; this is a problem because the temperature of the compartment has proven to be dependent on outside variables such as ambient atmospheric temperature and requires varying current in order to stabilize at 125°C. In other words, on some days the bar may need a slightly different amperage to achieve 125°C than other days, which makes the variac power supply a better option than a hard wired circuit of set voltage/current. Eventually a temperature controller with feedback will be installed to better control the temperature of the bar.

Electrically, Micro-VCM was wired for 208 V, 2-phase power because JPL was equipped with a 3-phase to 2-phase power transformer that the entire machine could be plugged into. Cal Poly does not have such a configuration, which resulted in the machine being broken up into multiple circuits/power cords; in other words, currently the machine is not a self-contained electrical unit with one power cord that feeds all of the components. Many other electrical aspects of the machine had to be analyzed, including trouble shooting the Brinkmann water-cooling unit, which had a break in its circuit, and the Welch mechanical pump, which was wired for 208 V instead of 115 V. Other changes included cleaning and replacing all O-rings on the machine and installing the gas feedthrough to allow venting of the chamber with nitrogen gas instead of air; ASTM E595 calls for venting the vacuum chamber with nitrogen instead of air in order to guard against material samples absorbing water vapor as they cool down from 125°C.

3.2 ATOMIC OXYGEN CHAMBER

Cal Poly's atomic oxygen simulation chamber is known as MAX, the Minimum Atmospheric eXperimentation chamber. MAX generates atomic oxygen by creating plasma and utilizing the

energy of the plasma to disassociate diatomic oxygen into atomic oxygen, much in the same way that VUV radiation has the energy to break O₂ bonds in space. Test chambers that utilize plasma to create AO are referred to as plasma ashers. Plasma ashers typically use RF energy to create plasma, and run on air or oxygen at pressures less than 200 mTorr.^[12]

MAX generates plasma that is specifically known as a capacitively coupled plasma, or a CCP. To create a CCP, two plates, or electrodes, are set up parallel to each other. One plate is powered with RF energy, while the other plate is grounded. When a plate is charged, plasma forms between the electrodes; this is known as an electrodeless discharge due to a plasma sheath that forms around the electrodes.^[12] This process is illustrated in the simple diagram shown in Figure 12, which captures the process taking place in MAX.

The RF electrode in MAX is a 15.25 cm diameter, 0.9 cm thick 6061 aluminum alloy disc. The electrode is placed inside a dark space shield in order to minimize secondary emissions off the electrode; the dark space shield improves the concentration of AO that occurs between the two plates. The ground plate is an aluminum square of dimensions 25.4 cm x 25.4 cm, and is placed 7.62 cm away from the powered electrode. A counter bored through hole exists in the center of the ground plate.

Figure 12. Simple schematic of capacitively coupled plasma system.^[12]

A Swagelok pipefitting is set in this hole from the bottom; the pipe is attached to a gas feedthrough from which air is bled into the vacuum chamber through a needle valve. This air bleed keeps the pressure of the chamber at ~ 175 mTorr, less than 200 mTorr as is typical of plasma ashers.^[12] The relatively high pressure is necessary to supply enough oxygen atoms to create AO. An aluminum cover plate with a #8 mirror finish is attached to the ground plate via eight screws. The cover plate has four evenly spaced holes in it for sample containment. Reference 5 dictates that samples should have a controlled area exposed to AO during testing, and they should be located at equal distances from the RF electrode.^[5] For this reason, the holes are precision machined to be 2.540 ± 0.003 cm in diameter which results in each test sample having 5.06 ± 0.02 cm² of exposed area during a test. Copper ground straps serve to ground the dark space shield and the grounded plate of the apparatus. Figure 13 shows how the electrode fits into the dark space shield, while Figure 14 shows a photograph of the entire apparatus assembled. Figure 14 shows both plates, with the top plate powered by an RF carrying coaxial cable wrapped in aluminum foil tape; the mirrored cover plate with sample containing holes are shown, as well as the copper ground straps.

Figure 13. Top, powered plate in MAX comprised of circular electrode and square dark space shield.^[12]

Figure 14. Assembled AO apparatus.

As previously mentioned, the AO apparatus in MAX requires RF power to be delivered to the electrode in order to create plasma. This is accomplished by implementing an RF power supply system that includes an RF generator, a load and matching network, and a system

controller. The RF generator is a Seren R301MKII; the generator operates at an RF frequency of 13.56 MHz and can supply up to 300 W of power. 13.56 MHz is the standard frequency set aside by the FCC for industrial plasma applications. For MAX, the Seren R301MKII is set at 125 W. The RF generator is paired with the Seren AT3 matching network which matches the impedance load of the plasma generator; the AT3 is necessary to eliminate any reflected signals produced in the load that could damage the RF generator. Finally, a Seren MC2 controller is matched with the AT3; the MC2 controls and adjusts the variable capacitors within the AT3 matchbox to automatically find a matching impedance and eliminate any reflected power.^[12]

Other than the RF power system, MAX is equipped with a Varian Dry Scroll vacuum pump, which is capable of maintaining a pressure of 175 ± 10 mTorr. Similarly to Micro-VCM, a Granville-Phillips 275 Convectron Gauge and Granville-Phillips 316 Vacuum Gauge Controller are used to monitor the pressure of the vacuum chamber. MAX is also equipped with a cryopump, ion gauge, and ionization gauge controller for experiments that require pressures on the order of $10^{-7} - 10^{-8}$ torr, but those items are not necessary for AO testing. A K-type thermocouple feedthrough must be attached to MAX during AO testing. These thermocouples are used to monitor the temperature of the ground plate during a test. This is because temperature affects the reaction probability of materials with AO, so if the ground plate gets too hot due to the plasma, AO test results will be incorrect; the established temperature to abort a test in MAX is 90°C. Finally, MAX is also equipped with a Hamamatsu L10706 deuterium lamp in order to simulate the VUV environment. VUV and AO have been shown to interact synergistically, and the addition of the lamp allows tests to be conducted in MAX to determine the relationship between the two. However, the lamp was out of commission during the testing conducted for this thesis and was not implemented. A full schematic of MAX is shown in Figure 15.

Figure 15. Full schematic for MAX chamber.

ASTM E2089^[2] is the standard set in place to ensure that AO erosion facilities yield reliable and comparable data. The standard outlines proper test procedures such as material preparation and proper weighing methods of samples, all of which is covered in Appendix B for MAX. The standard also establishes a method for determining the effective AO flux and fluence of an AO testing chamber. There are four materials that are understood well enough to use as witness samples to calculate the flux and fluence: Kapton polyimide (H or HN), TFE fluorocarbon fluorinated ethylene propylene (FEP), low density polyethylene, and pyrolytic graphite. Using the known reaction efficiency of one of these materials allows for the effective flux to be calculated using the equation,

$$f = \frac{\Delta M}{A \cdot \rho \cdot E \cdot t} \quad (11)$$

where f is the effective flux in atoms/cm²/s, ΔM is the change in mass in grams, A is the exposed area of the sample and is 5.06 ± 0.02 cm², ρ is the density in g/cm³, E is the reaction efficiency in cm³/atom, and t is the test time in seconds. The effective fluence can then be solved for by multiplying by the time of the test, or

$$F = \frac{\Delta M}{A * \rho * E} \quad (12)$$

where F is the effective fluence in atoms/cm². These equations were used to calculate the effective flux and fluence yielded by MAX for this thesis. The effective flux and fluence values are calculated with the in-space reaction efficiency of one of the four aforementioned materials. This means that Eq. 11 and Eq. 12 yield flux and fluence values that are extrapolated to in-space values, thereby designating the values as the “effective” flux and fluence. The effective flux and fluence values are not representative of the actual number of incident AO atoms yielded by an AO chamber, but instead are the equivalent in-space values that can be extrapolated from the test chamber. This is not to say the AO environment that test samples see in RF plasma chambers is equivalent to the AO environment experienced in space; it is extremely difficult for a ground-based AO system to duplicate the exact conditions experienced in orbital environments. Specifically, the interaction mechanisms between an AO plasma and test material include thermal energy atoms, high-energy ions, VUV light, excited-state neutrals, and isotropic AO impingement, all of which can affect the interactions between AO and a test material and result in erosion that is different from what is seen in space.^[23]

The AO plasma created in MAX is an isotropic, thermal plasma. The AO atoms created have an average energy of ~0.04–0.1 eV. While it is difficult to measure, it is estimated that the

AO plasma consists of roughly 90% AO and 10% ionized oxygen (Max Glicklin, personal communication, November 21, 2014). This AO plasma is different than orbital AO in several ways. First, orbital AO impacts spacecraft surfaces in one direction at high velocities. This directional impact on orbit results in pits and cones to form on the surface as material is eroded away. Since the AO in MAX is isotropic, the velocities of the AO atoms are omnidirectional, i.e. they are moving in all directions; this means that materials tested in MAX will not demonstrate the pits and cones that are seen from orbital erosion. Also, Laser detonation chambers capable of accelerating AO to orbital velocities are possible to build, but they are very expensive.^[19]

Another major difference between AO in MAX and orbital AO is the energy levels of the AO atoms. Orbital AO collides with spacecraft with an average energy of ~ 4.5 eV, so the average AO energy of 0.04–0.1 eV yielded in MAX is much less than the average energy seen on orbit. At first glance, it would appear that the AO generated in MAX would not have the energy levels required to break chemical bonds and oxidize materials. However, AO follows a Maxwellian distribution, which means that even though the AO in Max has an average energy of 0.04–0.1 eV, a small percentage of the atoms have energy levels that reach 4.5 eV and beyond. The way plasma ashers take advantage of the Maxwellian distribution is to create an extremely high number density of AO. Each incident AO atom that interacts with a surface material has a given probability of reacting with the material; the higher the energy of the AO, the higher the probability of a reaction. AO in MAX has a relatively low average probability of reaction; however, the extremely high number density of AO that MAX achieves results in enough AO atoms reacting with the test materials to accomplish erosion that is qualitatively similar to orbital erosion.^{[12][5]} The high number density and isotropic impingement can lead to accelerated rates of

undercutting, a phenomenon where AO atoms penetrate and erode a material at a defect site of surface coating.^[5]

The AO plasma in MAX also generates UV radiation, high-energy ions, and excited state neutrals (such as excited oxygen molecules). Each of these side affects can interact with test materials and result in physical and chemical changes that are different from the changes the material undergoes when it is exposed to solely AO. These constituents that are present in plasma are also present in the space environment; a spacecraft in orbit that is exposed to AO will also be exposed to UV radiation, high energy ions, and excited neutrals. However, materials are typically exposed to vastly larger quantities of the secondary constituents in plasma ashers than in space.^[23] For example, the flux of UV photons in plasma ashers can be thousands of times as great as the flux seen in space.^[20] This is problematic, as UV radiation has been shown to accelerate AO erosion; exposure to UV radiation in plasma ashers can result in more erosion and degradation than what would be seen on orbit. Similar instances can be seen with ions and excited neutrals, as plasma ashers create higher numbers of these charged species which can cause different surface interactions and erosion of materials.^{[23][20][22]}

In Reference 22, Miller, Banks, and Walters attempted to investigate the effects that different effective fluences, charged species, and UV radiation had on the erosion of materials when the materials were tested in an RF plasma AO chamber. In the study, different measures were taken to isolate the effects of each secondary constituent and establish the effect that each constituent had on the measured erosion of different materials. The study showed that Kapton H was affected by changing the AO fluence and slightly affected by charged species. FEP Teflon, however, did not exhibit much of a relationship with changing fluences, but was affected more by charged species and UV radiation. These are only the results of one study, but they show that

not only do ions, neutrals, and UV radiation affect AO test results, but separate materials are affected by each entity differently.^[22] This makes it extremely difficult to develop a set of standards that can be trusted to extrapolate RF AO plasma test results to in-space values. Individual materials need to be tested under isolated conditions in order to develop trends that can help to predict how each material will be affected by ions, neutrals, UV radiation, etc. Ascertaining these trends through testing will assist in creating a method for extrapolating in-space AO predictions from RF plasma testing.^[22]

ASTM E2089 does not define methods that will specifically account for the side effects of RF plasma testing. The intent of ASTM E2089 is to outline procedures that can be applied to a wide variety of test facilities in order to minimize the variability in results within specific facilities and understand the differences that are prevalent throughout different facilities. ASTM E2089 establishes that Eq. 11 and Eq. 12 should be used to determine the effective flux and fluence values that are yielded from a test chamber based on mass loss, but these values do not specifically take different levels of UV radiation and ions across different chambers into account in order to correct for differences in erosion mechanisms.^[1] There are publications, such as JPL's "Protocol for Atomic Oxygen Testing of Materials in Ground-Based Facilities"^[23] that attempt to categorize different AO test chambers based on how similar the AO reaction mechanisms will be to in-space AO exposure. The protocol breaks AO chambers into three levels. Level 1 testing facilities are classified as screening tests to determine how a material will react in orbital AO; synergistic effects such as ions and UV radiation are present but cannot be quantified. Level 2 facilities are capable of mitigating synergistic effects so that they either do not exist or are shielded against so that ions and UV radiation do not reach the test samples; level 2 facilities also involve average AO energies that are similar to orbital AO, as well as directional AO

impingement. Level 3 facilities are able to combine measurable synergistic effects with AO exposure in order to most accurately represent the interactions that happen in space. Under these criteria, MAX would be considered a level 1 test facility, which means it is capable of conducting screening tests in order to give a baseline understanding of how a material will react to AO in space.^[23]

The takeaway from this information is that the AO environment of MAX is different than that of the AO environment found in space, but MAX still serves as a legitimate test facility that is capable of testing how a material is likely to react when exposed to orbital AO. Kapton HN is a material with a well-understood in-space AO reaction efficiency. Testing Kapton HN samples in MAX allows Eq. 11 and Eq. 12 to be used to calculate the effective flux and fluence of AO to establish the equivalent space dosage that the chamber yields. AO is achieved through a plasma in Max, so omni-directionality, high-energy ions, excited neutrals, and UV radiation contribute to different reaction mechanisms and result in different levels of erosion and surface topography than what would be seen on orbit. That being said, materials that are unreactive to AO on orbit are unreactive in AO plasmas, and materials that are reactive to AO on orbit are reactive in AO plasmas.^[18] Materials tested in MAX undergo erosion that is qualitatively similar to orbital erosion, which makes MAX a viable testing chamber for this thesis.

4. MICRO-VCM & MAX VALIDATION TESTS AND RESULTS

4.1 MICRO-VCM VALIDATION

When Micro-VCM was donated to Cal Poly, it had not been used to collect outgassing data for years. The validity of the outgassing tests that were conducted before the apparatus was shut down at JPL was also called into question. For this reason, and because Micro-VCM underwent many changes during the refurbishing process, it was very important to perform outgas tests according to ASTM E595 and confirm that the apparatus was capable of collecting valid test data. It is important to validate Micro-VCM for two reasons. For one, Cal Poly's Space Environments Lab cannot claim the ability to conduct outgassing tests unless Micro-VCM is validated. Secondly, Micro-VCM would not be able to be used for this thesis to determine if AO exposure affects outgassing properties if the TML and CVCM values yielded by the apparatus could not be trusted.

The thought process behind validating Micro-VCM was to find and purchase a material that has known TML and CVCM values and see if testing the material yielded the correct values. NASA's outgassing database was used to search for materials with known outgassing values; another restriction was set in place to only use a material made by Sheldahl, simply because Cal Poly has a working relationship with Sheldahl and has purchased their materials in the past. A relatively high outgassing material was also desired to easily compare outgassing numbers. 1.0 mil thick Second Surface Aluminum Coated Polyimide Tape with Silicone Adhesive by Sheldahl was chosen and purchased for testing; according to NASA's database, the tape has a 1.98% TML and 0.38% CVCM. The intention was to also test Sheldahl's Silvered Teflon Film, which exhibited 0.00% TML and CVCM, but this material was not tested for reasons explained later.

The aluminum tape was prepared and tested according to ASTM E595; however, not all of the procedures from the standard could be followed for various reasons. A detailed, step by step procedure for the operations of Micro-VCM is given in Appendix B, but a summary is given here in order to highlight parts of the procedure from ASTM E595 that cannot be adhered to because of limitations of Cal Poly's facilities. Testing materials are to be kept in a desiccator until 24 hours before a test, which was accomplished with the aluminum tape. However, test samples are to be moved into a humidity and temperature controlled apparatus in order to be conditioned at 23°C and 50% relative humidity for 24 hours before being placed into Micro-VCM. A Tupperware container was equipped with Planet Waves Humidpaks, which are packs designed to absorb and give off moisture in order to keep an enclosed container at 50% humidity, in order to condition the samples at the correct humidity. However, designing a temperature controlled box would have cost money and time that was not available for this thesis; since 23°C is close to typical room temperatures, it was determined that it would be acceptable for the temperature of the box to be uncontrolled.

ASTM E595 establishes that the collection discs used to collect volatile condensable mass for CVCM testing shall be chromium plated discs. Copper discs were used for testing in Micro-VCM because chrome plated discs were not available for this thesis. Calculating TML and CVCM requires that test samples be weighed before and after the outgassing test; these mass measurements are supposed to be carried out using a microbalance with accuracy of $\pm 1\mu\text{g}$. Microbalances of this capacity are extremely expensive, as quotes came back at ~\$26,000. None of the facilities on Cal Poly's campus have access to such a fine microbalance. For this reason, a semi-microbalance from Cal Poly's Chemistry department, the Mettler-Toledo MS105DU, was

utilized for weighing test samples; the balance is capable of taking measurements on the order of 10^{-5} grams, but this still results in higher error values than industry standard ASTM E595 tests.

Also, test specimens that have completed a 24-hour outgassing test are to be removed from Micro-VCM and placed in a desiccator; the samples are then to be weighed within 30 minutes of removal. Since it was necessary to use a balance from the Chemistry department, the test samples had to be walked across campus to the lab that housed the balance. While measurements were taken as quickly as possible, the 30-minute threshold was not always met. Finally, ASTM E595 mandates that samples be kept at $125 \pm 1^{\circ}\text{C}$ in the copper sample compartment bar. A temperature gradient existed along the bar from top to bottom, as the top of the bar typically went to $\sim 126^{\circ}\text{C}$, while the bottom of the bar went to $\sim 124^{\circ}\text{C}$. The middle of the bar was measured at $\sim 128^{\circ}\text{C}$, meaning that a few compartments of the middle compartments were slightly outside of the $\pm 1^{\circ}\text{C}$ deviation set by the standard. These compartments were still utilized for testing in order to get the maximum number of samples tested.

Besides the caveats mentioned above, the aluminum Kapton tape was tested according to ASTM E595 in Micro-VCM; the results of three validation tests are shown in Table 3 and Table 4, with TML and CVCM calculations made according to Eq. 8 and Eq. 9. Table 3 and Table 4 show the average TML and CVCM values for each test, as well as the standard deviations of each test set and the acceptable standard deviations as mandated by ASTM E595. The acceptable standard deviations are discussed in greater detail later in this section. The third and final CVCM test does not have any data because the test was interrupted and the discs could not be weighed within a reasonable amount of time after the conclusion of the test.

Each of the three tests shown in Table 3 and Table 4 show a different number of samples because at the time, the proper procedures were still being experimented with to get correct

outgassing numbers. Also, ASTM E595 dictates that only three material samples need to be tested and averaged per test, but for validation tests it was decided to test as many samples as possible. A lot of time was spent trying to match the TML and CVCM numbers from tests in Micro-VCM to the 1.98% TML and 0.38% CVCM values found in NASA's database. The first preliminary tests that were conducted in Micro-VCM exhibited TML values that fell significantly short of the 1.98% target TML. These procedures followed ASTM E595's mandate that tapes be adhered to aluminum foil strips and placed in aluminum foil boats for testing. When this procedure failed to yield results close to NASA's numbers, the procedure was changed and the aluminum foil strips and boats were eliminated; instead, the aluminum tape was lightly rolled, as shown in Figure 16.

Figure 16. Rolled aluminum Kapton tape test specimen.

This rolling procedure resulted in the TML numbers shown in Table 3, which are extremely close to the 1.98% value from NASA's records. Upon further investigation, it was discovered that the TML and CVCM values recorded by NASA were taken from tests conducted in 1991. Since ASTM E595 has been updated many times, it is possible that the procedure followed by

techs for the 1991 test did not include aluminum foil strips and boats which is why the rolled tape procedure was able to get values so close to the target value of 1.98%.

While the TML values in Table 3 are very close to the expected value of 1.98% (within 3.0%, 0.5%, and 1.0% of the target value for tests 1, 2, and 3, respectively), the CVCM values in Table 4 are not at all close to the 0.38% expected value. One possible cause that was looked into was whether the copper collector discs had experienced contamination from other sources besides the test samples or whether cross contamination had occurred. The way to test this is to include a witness copper disc in the test that is placed in front of an empty test compartment. According to ASTM E595, if the witness collector disc experiences an increase of 20 μg or more, the collector disc most likely underwent poor preparation and cleaning; an increase of 50 μg or more means the chamber has a contamination problem that needs to be addressed. This is a problem for our equipment since the semi-microbalance implemented for this thesis cannot measure down to micro-grams; 20 μg (0.020 mg) and 50 μg (0.050 mg) is not even within the listed typical repeatability of the semi-microbalance (0.06 mg) at nominal loads of 100 mg. For this reason, the 20 and 50 μg guideline set by ASTM E595 cannot be followed until Cal Poly's Space Environments Lab gains access to a microbalance. However, witness samples were still used for every test conducted in Micro-VCM. The mass gain of each collector disc for test 1 and test 2, respectively, was 0.08 mg and 0.02 mg, which are values within the standard deviation of the semi-microbalance itself (using the repeatability listed on the balance of 0.06 mg, the subtraction of the two discs to get the mass gain results in an error of ± 0.12 mg), so they were not factored into the CVCM calculation as a correction factor.

Even if the witness discs were used as a correction in the CVCM calculation, the resulting CVCM values still would not be close to the expected 0.38% value. However, further

research and discussions with Tim O'Donnell revealed that there is often a high amount of variation in TML and CVCM values between different test facilities (Tim O'Donnell, personal communication, August 22, 2014). For instance, Figure 17 shows the variation between outgassing test results from test facilities at Johnson Space Center, Space Systems Loral, and the European Space Agency.^[28]

Agency	Johnson Space Center 313-686-11-04-01	Space Systems Loral July 1993	European Space Agency
Total Mass Loss	0.74%	0.012%	0.94%
Volatile Condensable Material	0.06%	0.002%	0%
Water Vapor Reabsorption	0.36%		0.38%

Figure 17. Table from Sheldahl document showing variation in outgassing data between test facilities for 3P adhesive^[28]

On top of the disparities shown in Figure 17, it was also shown that there are materials in NASA's database with ranges of TML and CVCM data since variability makes it very difficult, and even incorrect, to establish a sole value for either property. For instance, 3M 1245 Tape is listed as having a TML of 0.16 – 0.47% and a CVCM of 0.05 – 0.21% (Tim O'Donnell, personal communication, August 22, 2014).

Because of the new information presented about outgassing variability, it was decided that aiming to match values for specific materials within NASA's database was not the correct way to go about validating Micro-VCM. If there was that much variation between established, industry standard test facilities between outgassing data, it would likely be impossible to match numbers with NASA's database. Therefore, it was determined that the more important metric to judge the performance of Micro-VCM by would be the standard deviation between the individual test samples within each test. ASTM E595 outlines guidelines for acceptability of an

outgassing test, and the European standard, ECSS-Q-70-02A^[11], clarifies it. Essentially, a valid TML test will exhibit a standard deviation between each sample of less than 1/10 of the average TML percentage values. Similarly, a valid CVCM test's standard deviation will be less than 1/5 of the average CVCM value. Since TML and CVCM values can get very small, the minimum standard deviations for TML and CVCM are 0.05% and 0.03%, respectively.

The values in the final columns titled “Acceptable (1/10 test)” and “Acceptable (1/5 test)” in Table 3 and Table 4 correspond to the acceptable standard deviation for that respective test; the values shown in those columns are equal to 1/10 of the average TML or 1/5 of the average CVCM from that specific test. The preceding columns in the tables show the standard deviation associated with the respective test. In comparing the standard deviations to the acceptable values, Table 3 shows that test 1 and test 2 did not quite meet the 1/10 test, but were very close. However, test 3's standard deviation of 0.09 came in well below the acceptable value of 0.20, which shows that as the procedure was refined, the values yielded by Micro-VCM increased in validity. Table 4 shows that the standard deviations for CVCM tests 1 and 2 were within the acceptable values as established by the 1/5 test.

Table 3. Total Mass Loss Validation Tests - Aluminum Kapton Tape

Sample #	1	2	3	4	5	6	Average $\pm$ 0.12	Standard Deviation	Acceptable (1/10 test)
Test 1 TML %	2.01	1.88	2.17	1.63	-	-	1.92	0.23	0.19
Test 2 TML %	2.24	1.96	1.61	2.14	1.99	-	1.99	0.24	0.20
Test 3 TML %	2.04	1.81	2.05	1.95	2.00	1.90	1.96	0.09	0.20

Table 4. CVCM Validation Tests - Aluminum Kapton Tape

Sample #	1	2	3	4	5	6	Average $\pm$ 0.12	Standard Deviation	Acceptable (1/5 test)
Test 1 CVCM %	1.06	0.87	1.18	0.87	-	-	0.99	0.15	0.20
Test 2 CVCM %	1.08	0.69	0.86	1.15	1.03	-	0.96	0.18	0.19
Test 3 CVCM %	-	-	-	-	-	-	N/A	N/A	N/A

The values shown in Table 3 and Table 4 were deemed acceptable to move on and utilize Micro-VCM for combined AO/outgassing testing. The procedures of the combined testing will be described in further detail later in this thesis, but for now it will be mentioned that the procedures allowed for the collection of more outgassing data without the influence of AO. To reiterate, TML and CVCM values were collected from samples not exposed to AO, and those results are shown in this section to further validate the ability of Micro-VCM to collect viable outgassing data. Aluminum Kapton tape was retested, as well as an ITO-Kapton-Aluminum film and Aluminum-Kapton film, all Sheldahl products. Table 5 shows the TML and CVCM results from the three tests, as well as the respective standard deviations and acceptable standard deviations. The table also shows the mass gain experienced by the witness collector disc for each respective test. If the weight gain of the witness disc was significant, it was used as a correction factor for the CVCM calculation as was shown in Eq. 10; the witness was used as a correction factor for the first two materials, the aluminum tape and ITO-Kapton-Al, but not the Al-Kapton film because the mass gain of 0.05 mg was within the standard deviation, or repeatability, of the measurements taken with the semi-microbalance.

Table 5. TML and CVCM data taken with Micro-VCM for three materials

	TML%	TML St. Dev.	Acceptable St. Dev.	CVCM%	CVCM St. Dev.	Acceptable St. Dev.	Witness Gain (mg)
Aluminum Kapton Tape	1.65 ± 0.07	0.24	0.16	0.50 ± 0.06	0.22	0.10	0.26
ITO-Kapton-Al	1.21 ± 0.08	0.07	0.12	-0.02 ± 0.07	0.04	0.03	-0.13
Al-Kapton	0.88 ± 0.07	0.08	0.09	0.08 ± 0.07	0.04	0.03	0.05

The first thing to note from Table 5 is the TML data. The table shows that while the aluminum tape standard deviation did not pass the 1/10 test, the ITO-Kapton-Al and the Al-Kapton did pass. This is not a cause for alarm, as the 1/10 test is there for a reason; it is expected

in outgassing tests that sometimes the results will not be acceptable and have to be retested. Since the standard deviation of 0.24 is outside of the acceptable value of 0.16, it would have to be decided whether to retest the material or list the results with an asterisk. The standard deviations of the next two materials, 0.07 and 0.08, however, are well within the acceptable values of 0.12 and 0.09, respectively, which lends credence to the assertion that Micro-VCM is capable of collecting good, significant TML data.

Similarly to the TML data for the aluminum Kapton tape shown in Table 5, the CVCM data for the tape failed the 1/5 test, even after passing it in the validation tests. This further highlights the variability involved with outgas testing, and shows that it is not uncommon to have to retest a material. The next two materials got very close to passing the test, as they were within 0.01 to the limit of 0.03% set by ECSS-Q-70-02A. It is interesting to note that the aluminum tape standard deviation failed to fall within range of acceptable margins, and that the TML and CVCM values from the Table 5 test varied significantly from the values shown in Table 3 and Table 4 from the validation tests; the high standard deviation, as well as the deviation from the values from the validation tests, go hand in hand and show that if the test had been conducted solely to collect outgassing data, it should be redone (the test was not redone because it was conducted as part of the procedure to test AO effects on outgassing).

Another interesting note to make about the results shown in Table 5 is the collector discs for the ITO-Kapton-Al samples actually experienced mass loss instead of mass gain. According to ECSS-Q-70-02A, all collector discs are baked out at a temperature $> 125^{\circ}\text{C}$ at a pressure of $< 7.5 \times 10^{-6}$ torr in order to outgas all materials before being used to collect volatiles. The discs are also to be cleaned with acetone or an acetone/ethanol mix. While these cleaning steps were taken before each and every outgassing test conducted, it is likely that the mass loss experienced in the

ITO-Kapton-Al test is a result of poor cleaning. As mentioned previously, the collector discs are supposed to be chrome plated discs, not copper. The reason for the chrome plating is that it is much easier to clean, so it is possible that the copper surfaces of the discs contributed to the difficulty in cleaning them properly for that specific test.

Overall, the numbers shown in Table 3, Table 4, and Table 5 show that while incidents will occur that require a retest of materials, Micro-VCM is predominantly capable of taking significant TML and CVCM values. It should not be a concern that some tests did not pass the standard deviation test; results from an Italian test facility that is routinely audited by the European Space Agency showed that out of eleven conducted tests, nine were accepted, which shows that even industry standard facilities do not always achieve acceptable results.^[14] Furthermore, it is to be expected that data sets from Micro-VCM would fail the standard deviation test more often than typical industry tests because measurements are not being taken with a high precision microbalance. Cal Poly's Space Environments Lab can move forward with confidence in conducting outgassing tests in Micro-VCM. In the future, Cal Poly can look into more ways to validate the Micro-VCM system; these methods (including the method of auditing outgassing chambers) are discussed in the future work section of this thesis.

4.2 MAX VALIDATION

MAX did not have to undergo nearly the same level of validation tests as Micro-VCM as MAX had already been validated immediately after its completion in 2012. However, since its first validation tests, MAX had been disassembled and undergone several changes, so it was necessary to conduct tests upon reassembly to establish the flux and fluence numbers that should be expected from it.

When MAX was reassembled to begin testing for this thesis, new coaxial cables were purchased and implemented to RF power from the power supply to the electrode, and a BNC feedthrough was implemented. Validation tests were conducted with the new configuration using samples of HN Kapton prepared according to ASTM E2089. The flux and fluence values from the tests were calculated using Eq. 11 and Eq. 12, using a density of $1.435 \pm 0.002 \text{ g/cm}^3$ and reaction efficiency of $3.00 \pm 10^{-24} \text{ cm}^3/\text{atom}$. The average flux between 7 samples was $1.260 \pm 0.142 \times 10^{16} \text{ atoms/cm}^2/\text{s}$ and the fluence for a 24 hour test was $1.088 \pm 0.108 \times 10^{21} \text{ atoms/cm}^2$. Witness Kapton samples were tested alongside other material samples throughout the combined AO/outgassing tests and compared to these values to ensure the flux and fluences experienced during the tests were on the same order of the validation tests. Compared to the original fluence value of $1.47 \times 10^{21} \text{ atoms/cm}^2$ from MAX's original validation tests, these numbers are a little low but still on the same order of magnitude. Witness samples (showed in the results section of this thesis) showed fluences closer to the original validation value.

5. COMBINED ATOMIC OXYGEN AND OUTGAS TESTING

There is good reason to wonder if AO exposure affects the outgas properties of materials. As previously mentioned, it has been shown that UV radiation increases the amount of outgassed contaminants from a material.^{[17][18]} AO interacts with materials in a similar way to UV radiation, as AO has the energy to break chemical bonds; AO goes a step further in being able to oxidize surface atoms. One study did show a relationship between AO and outgassing, as AO exposure was shown to slightly reduce outgassing of an O-ring.^[17] It is possible to visualize all three mechanisms of outgassing being affected by AO. The breaking of bonds and oxidation that occurs on material surfaces due to AO could change the amount of material desorbed from the surface. The erosion of material due to AO could allow more or less trapped gases to diffuse to the surface and be outgassed. Since decomposition is essentially the outgassing of the degraded material itself, the degradation of material through AO erosion could lead to more outgassed material through decomposition. It is possible that any of these mechanisms could be affected in a way to outgas more or less material than is typical, or there could be no relationship between AO and outgassing at all.

Because of the lack of knowledge of the relationship between AO and outgassing, and the different possibilities AO affecting different outgassing mechanisms, two testing procedures were established for this thesis to examine the effects between AO and outgassing. The first procedure aimed to simply expose a set of samples to AO and compare their outgassing to a set of control samples. The second procedure involved outgassing a full set of sample according to ASTM E595 first, then exposing them to AO, and then outgassing them according to ASTM E595 once again after AO exposure. This procedure aimed to examine whether AO exposure allowed a material to outgas more or less after already being outgassed once; the theory was that

it would be possible for AO to erode the material and its layers to allow more trapped gases to escape during the second, post-AO, outgassing test that had not escaped during the first outgassing test.

While the outgassing procedures of ASTM E595 and the AO procedures of ASTM E2089 are outlined in Appendix B, the procedures to conduct combined AO and outgassing tests are discussed in the following pages. The steps taken in the first procedure are outlined in Figure 18.

Figure 18. Block diagram for steps taken in procedure 1, AO - outgas.

As Figure 18 shows, the procedure begins with preparing 9 total material test samples. A total of 9 samples was chosen because Micro-VCM has the capability of testing 9 samples at once. The 9

samples were prepared according to ASTM E595, meaning the samples were conditioned at 50% humidity for 24 hours and then weighed. The diagram also shows that a witness sample was prepared; this witness sample is a Kapton HN sample that is outgassed for 48 hours as outlined by ASTM E2089. The witness sample is very important as it was placed in MAX during the AO testing phase of the procedure and used to calculate the flux and fluence of AO that the material test samples were exposed to during the AO testing. Next, the 9 samples were broken up; 6 samples were placed in MAX for a 24-hour AO test, and 3 control samples were placed in Micro-VCM. The decision to place the 3 control samples in Micro-VCM was very important. When MAX is running, the ground plate that the test samples sit on is heated to $\sim 60^{\circ}\text{C}$, and the pressure within the chamber is ~ 175 mTorr. This means that as test samples in MAX are being exposed to AO, they are also being subjected to a temperature pressure that will instigate outgassing. This is why it was decided to put the 3 control samples in Micro-VCM as the other 6 test samples were being exposed to AO. The 3 control samples in Micro-VCM were raised to a temperature of 60°C and the pressure in the chamber was kept at ~ 175 mTorr in order to outgas them under the same conditions that the 6 test samples were at in MAX. In this way, the only difference between the control set and AO set of samples was the exposure of AO. Had the intermittent step of placing the control samples in Micro-VCM not been taken, the argument could be made that the 6 test samples were outgassed more than the control set and that it would be unjustifiable to compare the two sets of samples at the end of the procedure.

After the 6 test samples and witness sample were tested in MAX and the 3 control samples were outgassed in Micro-VCM, the samples were removed and weighed; the CVCM collector discs were also weighed at this time. Directly after weighing, all 9 samples were then placed in Micro-VCM to be outgassed according to ASTM E595 i.e. at 125°C and $<5 \times 10^{-5}$ torr

for 24 hours. Upon removal, all 9 samples and the copper collector discs were weighed once again so that the TML and CVCM values from the AO set and control set could be averaged and compared.

The second procedure was carried out similarly to the first, and is illustrated in Figure 19.

Figure 19. Block diagram for procedure 2, outgas-AO-outgas.

In the second procedure, the samples were outgassed, exposed to AO, and then outgassed again. The procedure started by preparing 9 samples and a witness sample just like in procedure 1. Procedure 2 deviated from procedure 1 after preparation; instead of placing samples into MAX for AO exposure, all 9 samples were placed in Micro-VCM and outgassed according to ASTM E595. After the initial outgassing for 24 hours, the samples were then broken up into a test set

with 6 samples and a control set with 3 samples and then placed in MAX and Micro-VCM respectively. Just like in the first procedure, the control set was outgassed at 60°C and ~175mTorr in order to isolate AO as the only variable in the test. Finally, the samples were all brought back together and outgassed according to ASTM E595 after the AO exposure. TML and CVCM values were calculated for both sets in order to make comparisons.

It is important to note that because of the nature of testing the same samples in MAX and Micro-VCM, it was necessary to make adaptations to the way material samples were situated. The way each test apparatus is set up (MAX and Micro-VCM) is not conducive to testing the same samples in both systems. MAX is designed to place a maximum of four 2" x 2" samples (3 test samples + 1 witness sample) in between the ground plate and a cover plate. The cover plate has four 1-inch diameter holes in it that expose the same area of the samples to AO; the holes are spaced identically so that all of the samples have the same area exposed at the same distance from the electrode. Micro-VCM, however, has a copper sample compartment bar with cylindrical compartments that are only ~0.625 inches in diameter and ~0.4375 inches deep. Essentially, Micro-VCM is designed for much smaller sample sizes than MAX is. Typically, samples of film or tape can be cut and placed in an aluminum boat in order to fit inside Micro-VCM's sample compartments. However, cutting pieces small enough to fit would have made it all but impossible to test the samples in MAX. It was also desired to place more than the typical three samples into MAX.

Because of the constraints of both systems, it was decided to cut long strips of material and roll them up to fit inside Micro-VCM's sample compartments; the strips were cut to be between 100 – 300 mg as is required by ASTM E595. This meant that the holes in MAX's cover plate could not be utilized, and instead the long sample strips were laid on top of the cover plate.

The result of laying the strips across the plate was that each sample was not exactly the same distance away from the electrode, and there was not a controlled area of exposure for each sample. This is not ideal, but since the goal of this thesis is to explore whether AO exposure affects outgassing properties, it was decided that this method sufficed to expose materials to AO. Had the goal been solely to see how AO affected materials, it would have been much more important to keep the test samples equidistant from the electrode with a controlled exposed area. Figure 20, Figure 21, and Figure 22 show the different samples as they were placed in MAX; all samples were rolled and placed in Micro-VCM. At times, the rolling of the samples resulted in the need to use tape to keep the samples flat in MAX; the tape was folded over on itself to ensure that no adhesive touched the samples to contaminate them. This is shown in Figure 22.

Figure 20. Rolled aluminized Kapton tape samples as they were placed in MAX.

Figure 21. ITO-Kapton-Al samples as they were placed in MAX.

Figure 22. Aluminum coated Kapton samples as they were placed in MAX.

6. RESULTS

Three Sheldahl materials were chosen to test for a relationship between AO exposure and outgassing: Second Surface Aluminum Coated Polyimide Tape with Silicone Adhesive (aluminum coated 1.0 mil Kapton Tape, Part #146490-001), ITO Coated Aluminized Polyimide (2.0 mil Kapton Film coated with ITO on one side and aluminum on the other, Part #146633-003), and Aluminum Coated Polyimide (2.0 mil Kapton film coated with aluminum, Part #146448-005). The aluminum coatings for all three materials were 1000 Å thick. For testing, the aluminum Kapton tape was rolled so that the Kapton side faced the AO. The ITO-Kapton-Al film was tested so that the ITO side was exposed to AO. The aluminum Kapton film was tested so that the aluminum side faced the AO since the aluminum Kapton tape had already been tested on the Kapton side. Each material was tested according to Procedure 1 (AO – CVCM) and Procedure 2 (CVCM – AO – CVCM) that were described in the previous section.

The results for all three materials after undergoing both procedures are shown in Table 6 and Table 7, as well as Figure 23 and Figure 24. TML and CVCM values for each procedure are shown in Table 6 and Table 7, along with the accompanying error values. The tables also show the mass gain experienced by the witness collector disk, and the AO fluence experienced during each test. If the mass gain of the witness collector disc was significant, i.e. outside of the error associated with the measurement, then the mass gain was used as a correction factor for CVCM calculations according to Eq. 10; only the aluminum Kapton tape calculations from Procedure 1 used the correction factor since all of the other collector discs experienced insignificant mass gain/loss. Figure 23 and Figure 24 show charts to supplement the tables of values; the charts serve as an illustration to directly compare the outgassing results of the control set of materials

with the AO set of materials. The charts include error bars as a visual to show how significant error was in the calculations.

The error calculated in this thesis was found knowing the errors are randomly and normally distributed. For TML and CVCM measurements, the mass of each material sample and collector disc was measured three times. The final mass that was used to calculate the TML or CVCM for that specific sample was the average of the three masses that were found with the semi-microbalance. This method accomplished two things. Averaging the mass from three measurements results in getting a final sample mass that is more representative of the actual mass loss that took place. Taking three mass measurements also allows for the standard deviation of the measurements to be calculated. This standard deviation was used as the uncertainty of the semi-microbalance in error calculations. The repeatability listed on the semi-microbalance of 0.06 mg could have been used for the uncertainty, but the calculated standard deviations were routinely greater or less than the listed repeatability; typical standard deviation values were 0.06 mg, but values also ranged from 0.04 mg – 0.09 mg. Therefore, using the standard deviation resulted in more realistic error calculations. Taking five mass measurements for each sample instead of three was experimented with, but it was determined that the standard deviations of the five measurements were very close to the standard deviations of the three measurements; because it was ideal to weigh the samples as quickly as possible, and the standard deviations were very similar between three and five, it was decided to take three measurements instead of five.

It is also important to note that the method of weighing the samples three times each was only adopted after the first material (the aluminum Kapton tape) had been tested and weighed; therefore, the error calculations for the aluminum Kapton tape use the listed repeatability of the

semi-microbalance (0.06 mg) as the uncertainty. Also, the standard deviations of each material were averaged to find the uncertainty value that would be used in the error calculation. In other words, nine test samples were measured three times each for each test, yielding nine standard deviations, one for each sample; those nine standard deviations were averaged to find the overall uncertainty of the semi-microbalance for that set of samples. Finally, it is important to note that the TML and CVCM values and their associated error values reported in Table 6 and Table 7 are all percentages of the initial mass. This means that when a value is given, such as 1.35 ± 0.12 for the control TML in Table 6, it means that the percent mass loss for that material was between 1.23 – 1.47%. To reiterate, the $\pm 0.12\%$ does not mean 0.12% of 1.35, but it is a value that can be added and subtracted to the value of 1.35 directly to find the range of TML calculated for that material.

The values shown in Table 6 and Table 7, and therefore the data shown in Figure 23 and Figure 24, are average TML and CVCM values. For example, the control TML that is reported in Table 6 and Figure 23 for the aluminum Kapton tape is the average TML value from all three of the control samples that were tested; similarly, the AO TML value is the average of the six AO samples that were tested. Note that although the error bars in Figure 24 appear to be larger than those in Figure 23, they are actually of the same magnitude; the scaling of Figure 24 is different than that of Figure 23, which makes the error bars look different.

Analyzing the values shown in Table 6 and the visuals in Figure 23 shows that not much of a trend can be gleaned from the data between the control set of tests and the AO set. The control and AO TML values for the aluminum Kapton tape are spot on with each other at $1.35 \pm 0.12\%$. The CVCM values differ, with the control set CVCM value being $0.81 \pm 0.12\%$ and the AO set being $0.63 \pm 0.12\%$; however, Figure 23 shows that the difference between the two

CVCM values is inside of the error bars. Figure 23 also shows that without even looking at the numbers from the ITO-Kapton-Al test sets, none of the calculated values are statistically significant as they all lie well within their respective error bars.

Figure 23 does show an interesting phenomenon that occurred with the aluminum Kapton film; while the CVCM of the film was insignificant for both control and AO sets, the material samples actually experienced a significant mass *gain* after undergoing outgas test conditions of 125°C and $\sim 2.0 \times 10^{-6}$ torr for 24 hours. This oddity is discussed in further detail later in this section. It is also important to note that during the AO testing of the aluminum Kapton, arcing was observed in between the electrodes of MAX. While the issue of arcing in MAX was eventually fixed, it was not fixed in time to retest the material.

Examining the results from Procedure 2 in Table 7 and Figure 24, it can be seen that the control and AO TML and CVCM values for the aluminum Kapton tape are almost identical to each other. No trend between the control and AO values can be observed. However, moving on to the ITO-Kapton-Al film shows some interesting results. The control and AO CVCM numbers for the ITO-Kapton-Al film are not significant as Figure 24 shows the values to be well within the error bars. The TML values, however, show a large difference. The control TML is $-0.03 \pm 0.09\%$, while the AO set experienced a CVCM value of $0.19 \pm 0.08\%$. Figure 24 shows graphically that the difference between the control value and AO value is significant as the difference lies outside of the error bars. This result shows that there is a possibility that AO exposure affected the mass loss experienced by the ITO-Kapton-Al. Moving on to the aluminum Kapton film, there is no significance between the control set and AO set, but there is the same startling result of the samples gaining mass, similarly to what happened to the material samples

Table 6. Outgassing results for three tested materials subjected to AO in MAX and then tested in Micro-VCM (Procedure 1).

	Control TML%	AO TML%	Control CVCM%	AO CVCM %	Witness Gain (mg)	AO Fluence (atoms/cm ²)
Al. Kapton Tape	1.35 ± 0.12	1.35 ± 0.12	0.81 ± 0.12	0.63 ± 0.12	0.35 ± 0.12	1.27 ± 0.128 × 10 ²¹
ITO-Kapton-Al	-0.01 ± 0.09	-0.07 ± 0.08	0.07 ± 0.08	0.07 ± 0.08	-0.06 ± 0.14	1.42 ± 0.133 × 10 ²¹
Al. x Kapton	-0.18 ± 0.08	-0.28 ± 0.09	-0.08 ± 0.07	0.02 ± 0.08	-0.01 ± 0.12	1.15 ± 0.125 × 10 ²¹

Figure 23. Column graph showing both TML and CVCM values for control and AO sets of data from Procedure 1.

Table 7. Outgassing results for three tested materials subjected to outgassing in Micro-VCM, AO in MAX, and then tested in again in Micro-VCM (Procedure 2).

	Control TML%	AO TML%	Control CVCM%	AO CVCM %	Witness Gain (mg)	AO Fluence (atoms/cm ²)
Al. Kapton Tape	0.27 ± 0.06	0.29 ± 0.07	0.26 ± 0.06	0.26 ± 0.07	0.12 ± 0.12	1.50 ± 0.135 × 10 ²¹
ITO-Kapton-Al	-0.03 ± 0.09	0.19 ± 0.08	-0.01 ± 0.10	-0.03 ± 0.08	0.14 ± 0.14	1.29 ± 0.129 × 10 ²¹
Al. x Kapton	-0.10 ± 0.07	-0.09 ± 0.09	0.04 ± 0.07	0.07 ± 0.09	0.08 ± 0.09	1.33 ± 0.130 × 10 ²¹

Figure 24. Column graph showing both TML and CVCM values for control and AO sets of data from Procedure

in Procedure 1. The TML value for the AO set did not extend outside the error bounds, but the value for the control set did; the trend of mass gain was clear and needed to be explored.

Two materials in different procedures exhibited a relationship between AO and an outgassing change. In Procedure 1, where materials were exposed to AO and then outgassed, the AO – exposed aluminum Kapton tape exhibited a lower CVCM value than the control set did, albeit the difference was inside of the error bounds for the values. One caveat to the difference lying outside of the error bounds is that the error for the aluminum Kapton tape was calculated using the typical value of repeatability on the semi-microbalance. This resulted in higher error than the other calculations because the standard deviations calculated for other materials were often slightly lower than the listed repeatability. Had the error been calculated with standard deviation values, the difference between the CVCM values may have actually lied outside of the error bounds; therefore this result will not be treated as completely insignificant such as the other tests, since it did at least display a noticeable trend between the AO and control set of samples. Also, remembering the study from Reference 7, researchers did not discount the trend that they saw simply because the difference lied within the error bars.^[7] While a concrete conclusion cannot be drawn from such a result, it is worth discussing as an observed trend.

The other material that exhibited a possible correlation was the ITO-Kapton-Al from Procedure 2, where materials were outgassed, exposed to AO, and then outgassed again. The upper bound of the control set of the material for TML was 0.06%, while the lower bound of the AO set was 0.11%, which shows that the AO set outgassed significantly more than the control set. One might expect a material that exhibited this much of a difference in Procedure 2 to experience the same or a similar difference in Procedure 1; however, oddly enough, there was no significant outgassing from the ITO-Kapton-Al in Procedure 1. Similarly, the difference

observed between the CVCM values of the aluminum Kapton tape in Procedure 1 was nowhere to be seen in Procedure 2.

Because the differences between the AO and control sets occurred in isolated incidents with these materials, and because of the overall magnitude of error in all of the TML and CVCM calculations for this thesis, the differences witnessed between the outgassing numbers for the aluminum Kapton tape and the ITO-Kapton-Al are not enough to conclude that AO exposure affects the outgassing properties of materials. While differences were seen in these two specific tests, all of the other tests conducted for this thesis showed no relationship between AO and outgassing. What the results do show is that more research needs to be done on this topic. The two materials in question should be retested to see if the results can be recreated, preferably using a full microbalance to take mass measurements, which would vastly reduce the error involved with the TML and CVCM calculations. Testing should also be expanded to research other materials; just because many of these tests did not exhibit any effect between AO and outgassing does not mean other materials will not.

Another aspect of the results that would call into question any definitive conclusions made is the aforementioned mass gain experienced by the aluminum Kapton film. While the aluminum Kapton film was the only material to experience significant mass gain (i.e. the only material to gain mass outside of the error bounds), examining Figure 23 and Figure 24 show that a surprising number of tests ended up showing mass gain. While these were not statistically significant, the trend of mass gain is still there. Also, a few intermediate tests showed mass gain as well; for instance, the ITO-Kapton-Al control set from Procedure 2 experienced slight mass gain after being baked at 60°C under ~175mTorr in Micro-VCM. All of these occurrences led to

the need to explore why material samples might be experiencing mass gain instead of mass loss during outgassing testing.

The aluminum Kapton film was retested with a slightly different procedure to see if mass gain could be eliminated. ASTM E595 says that while cleaning the apparatus in Micro-VCM with acetone and ethanol after every use is necessary, the chamber does not always have to be baked out. The chamber only has to be baked out at a temperature of 150°C if there are anomalous mass gains on the witness collector discs, or at least once every four months. For this reason, the chamber was baked out once a week (it took one week to get through one test material for one of Procedures 1 & 2). The chamber was not baked out after every use because it was determined that it would be ideal to move the samples through the procedures without any extra downtime sitting in the desiccator; although the desiccator is mostly devoid of water vapor, samples still reabsorbed small amounts of material while in the desiccator. However, it is possible that because the chamber was not baked out after each and every use, contaminants built up on the apparatus and adsorbed onto the surfaces of the material samples that experienced mass gain.

A new test of the aluminum Kapton film was conducted, following Procedure 1 but with several differences. First, at this point in time, MAX was still arcing between the plates and could not be used for testing. This means that nine samples were placed in Micro-VCM and baked at 60°C at ~175 mTorr to replicate the procedure the control samples from Procedure 1 were put through. The loss of MAX for this test did not matter because the control set from Procedure 1 exhibited mass gain just like the AO set. After the nine samples were baked out, they were weighed and placed in the desiccator instead of being placed directly back into Micro-VCM for testing as was done in Procedure 1. The samples remained in the desiccator for 24

hours while Micro-VCM was baked out at 150°C. After the bake out, the samples were placed back into Micro-VCM to be tested according to regular ASTM E595 procedures. The results of this test are shown in Figure 25; the bar graph in this figure includes the data for the aluminum Kapton tests from Procedure 1 that exhibited mass gain for a comparison.

Figure 25. Results from mass gain investigation test compared with the Al. Kapton results that experienced mass gain from Procedure 1.

The results from the investigation test, labeled “new set” and shown on the right of the bar graph in Figure 25, are clearly different from the TML values of the Al. Kapton from Procedure 1. The new samples that were kept in a desiccator while the chamber was baked out experience ~0.35% mass loss, a stark contrast to the previous samples which experienced ~0.20% and ~0.30% mass gain. While only one test was conducted, the results point to the likelihood that the cause of mass gain for certain sample sets in this thesis was from contaminants that were eliminated when the chamber was baked out. One reason that contaminants were more plentiful than expected in the apparatus could be the 60°C tests that were conducted prior to the final outgassing test that the materials experienced mass gain in.

Remembering Eq. 5, the lower the temperature of a surface, the more time a contaminant will stay on it. It is possible that products that outgassed during the 60°C exposure collected on parts of the apparatus that are hard to clean. Those products could have then left the surfaces and adsorbed onto or absorbed into the material samples, causing mass gain. It is important to note that while the new test samples were in the desiccator, they did experience mass gain; this extra mass could have been outgassed during the test, offsetting any mass gain they may have been experiencing. However, the average mass gain across the test set was ~0.54 mg, whereas the average mass loss after the final outgassing test was ~0.52 mg. This means that even though the mass gain from the desiccator helped to offset the final TML value, the TML for the test still would not have experienced any mass gain in the final outgassing test, it would have instead displayed ~0.00% TML, which makes much more sense than mass gain. Essentially, it is unknown whether mass gain was due to a lack of bake out process, but it is a possibility. More tests will likely illuminate the correct test and cleaning procedures to avoid contamination and mass gain problems.

7. CONCLUSION

A Micro-VCM apparatus was refurbished for Cal Poly's Space Environments Lab. The apparatus was validated with outgassing testing according to ASTM E595 and was then used to determine if atomic oxygen exposure has any effect on the outgassing properties of materials.

Great measure was taken to restore Micro-VCM to full functionality. Once the entire chamber was ready to create the conditions mandated by ASTM E595 for outgas testing (125°C sample compartment bar, 25°C collector discs, and $<5 \times 10^{-5}$ torr for 24 hours), validation tests were conducted to establish that the apparatus was capable of collecting TML and CVCM data. Aluminum Kapton tape was purchased to conduct validation tests. The tape was chosen because it has values of 1.98% TML and 0.38% CVCM in NASA's database; in the beginning of testing it was thought that making a comparison to specific materials was ideal for validation, and materials that outgassed more rather than less were ideal for comparisons. Tests with the tape were conducted and while a procedure was found that yielded TML values close to the target of 1.98%, the CVCM results did not match.

Further research illuminated the great amount of variation that is typical between outgas test facilities. It was then decided that due to variation in outgassing numbers, matching results to values in NASA's database was not the best way to validate the system. It was decided instead to focus on the standard deviations of the set of test samples; ensuring the standard deviation of the TML values is less than 1/10 of the average TML and the standard deviation of the average CVCM is less than 1/5 of the CVCM value itself is part of ASTM E595 as a check for whether or not to accept the test or not. While the values collected from Micro-VCM did not always pass this test, it did often enough to warrant moving on to combined AO and outgassing testing. In the

future, if the opportunity arises to test materials in tandem with an established facility (such as at JPL), it should be taken to attempt to see how much the results vary.

Two procedures were developed to investigate whether AO exposure has any effect on the outgassing of materials. The first procedure involved exposing samples to AO using Cal Poly's atomic oxygen simulator, MAX, and then placing them in Micro-VCM. AO test samples were compared to a control set of samples; measures were taken with the control set to ensure that AO exposure was the only different variable between it and the AO exposed set. The second procedure involved outgassing samples in Micro-VCM according to ASTM E595 first, then placing them in MAX for AO exposure, and finally putting them back in Micro-VCM for a final outgas test. This procedure aimed at investigating whether AO exposure allowed materials to outgas more or less after they had already been outgassed once. Just like Procedure 1, Procedure 2 included comparing a control set of samples to an AO set.

Three materials underwent testing under both procedures, aluminum Kapton tape, ITO-Kapton-Al film, and aluminum Kapton film. Three materials undergoing two procedures results in a total of six tests conducted. Of the six tests, two showed a difference between the AO set and the control set of samples. The AO exposed aluminum Kapton tape from Procedure 1 showed a decrease in CVCM when compared to the control set, however, the difference lied inside of the error bounds. The AO exposed ITO-Kapton-Al exhibited a far greater TML than the control samples did; this was the most significant observed difference, and the difference lied well outside the error bounds for the TML calculations. Other than those two tests, all the other tests resulted in no difference between the AO set and control set, or an insignificant difference because of large error bars.

While two materials exhibited a change in outgassing after AO exposure, two test results are not enough to make an overarching conclusion surrounding AO's effects on materials or to develop trend lines that can help to better understand the phenomenon. The error involved in the calculations is very high due to the use of a semi-microbalance instead of a full microbalance, which renders many of the results inconclusive. Even though two tests showed a change between AO exposed samples and control samples, one test did so by a relatively small margin that was undermined by large error bars. Both materials should be retested multiple times to see if the results can be recreated. Future tests would ideally be conducted using a microbalance, which has the precision to pick up on the fine changes that could be taking place between samples exposed and not exposed to AO. Also, many more materials need to be tested; the materials available to Cal Poly's lab limited this thesis, but it is possible that other materials, especially multi-layered materials, could respond much differently to AO and demonstrate solid relationship between AO and outgassing. Furthermore, as already highlighted in the motivation of this thesis, these results cannot be directly extrapolated to predict in-space behavior of materials that are simultaneously outgassing and being exposed to AO. This is because the testing of this thesis involved asynchronous outgassing and AO exposure, which does not reflect the actual interaction that would take place on orbit.

The testing and theories provided in this thesis established that it is very possible that AO could affect outgassing properties since two materials' outgassing properties were shown to respond to AO. More tests must be conducted before a stronger conclusion can be made. The work done on Micro-VCM to get the apparatus up and running for this thesis gives Cal Poly's Space Environments Lab a viable mechanism to test materials for TML and CVCM values. Micro-VCM can be used to continue exploring the effects of AO on outgassing properties, or to

conduct outgassing tests on other materials without investigating AO's effects. While Micro-VCM has been proven to produce acceptable results, the next step in the process should include working with an established facility to further validate its ability to collect TML and CVCM data.

8. FUTURE WORK

8.1 MICRO-VCM

There are many aspects of the Micro-VCM apparatus and the accompanying procedures that can be updated and improved upon. First, the copper CVCN collector discs should be plated in chromium. Chrome plating will be much easier to clean than the copper surfaces of the discs after a test and ensure they are clean of all contaminants prior to a new test. It will also keep the copper from undergoing oxidation and other reactions with atmospheric constituents. An attempt to get the copper discs chrome plated was made during the course of this thesis, but it proved difficult to find a vendor who could carry out the process. The reason it is a difficult process to plate the discs with chrome is they are so small and thin; a lot of force will be placed on the discs when they are racked for the chrome plating process. Not much time was spent trying to find a vendor for the chrome-plating project, so it is possible that there are many vendors out there with the ability to easily plate the discs and they just did not turn up in the quick search. If it proves difficult to find a vendor, Tim O'Donnell of JPL might have an idea of where to go because he is very familiar with the plating process; JPL may have a connection since they need collector discs for their own outgassing apparatus. In the meantime, the copper discs can also be cleaned with vinegar to remove the top layer of tarnish; this extra step might help reduce any contamination problems.

Currently the copper sample compartment bar is heated using a variac power supply that is hooked up to the resistor that is soldered to the copper bar. While this method works to heat the bar to 125°C, it is not ideal because the temperature has to be manually controlled by adjusting the voltage on the power supply. This would not be a problem if the voltage could be

set at a constant value to maintain the temperature of the bar, but this is not the case; the voltage needed to keep the bar at $\sim 125^{\circ}\text{C}$ fluctuates with atmospheric conditions. Sometimes if the power supply is left unattended to, the temperature of the bar will rise or fall even at a constant voltage/current. To circumvent this problem, an Arduino was purchased and a temperature controller was designed and put together by Ryan Moskaluk. The controller is capable of using feedback to adjust the supply of current through the resistor and thereby regulate the temperature of the copper bar to 125°C . The controller is also equipped with an LCD screen to display the actual temperatures of the copper bar and the cold plate. The temperature controller was not completed by the conclusion of this thesis, but the electronics are in place and just need to be assembled in a box, which can then be installed on the Micro-VCM machine and fine tuned.

Another aspect of the copper containment bar that can be looked into is the lack of uniform heating along the bar. As was previously mentioned in the validation section of this thesis, it has been shown that a temperature gradient exists along the compartment bar from the top of the bar to the bottom. The top of the bar typically goes to $\sim 126^{\circ}\text{C}$, then the temperature rises down the bar until it reaches a maximum temperature of $\sim 128^{\circ}\text{C}$ at the middle of the bar, and then the temperature decreases down the bar to get to 124°C at the very bottom. There are enough thermocouples on Micro-VCM to measure the temperature along the entire bar and find out what temperatures each compartment is going to (this has been done before but only the top, middle, and bottom numbers are reported). It should be noted that heating up the bar slowly (i.e. less current/ lower voltage from the variac power supply) results in a much tighter temperature gradient. The temperatures of each compartment, when the bar is heated slowly to $\sim 125^{\circ}\text{C}$, should be explored. Once differences in temperature are established, it can be decided whether certain compartments should be eliminated compartments that can be used to put samples in. For

instance, it might turn out that all of the compartments go to $\sim 125^{\circ}\text{C}$ within $\pm 1^{\circ}\text{C}$ except for two middle compartments which might be outside of 125°C by 3°C or 4°C . Discussions with Dr. A and Tim O'Donnell and more research could help to determine whether those compartments should be eliminated as test compartments. Multiple tests can also be performed to try and determine if the extra few degrees of the compartments have any effect on the TML/CVCM values of materials. The same material can be tested in a compartment at $\sim 125^{\circ}\text{C}$ and in another compartment at $\sim 128^{\circ}\text{C}$ (or whatever temperature it is found to go to) multiple times. The averages of the TML and CVCM values from each compartment can be compared to see if there is any statistical difference between the two sets of data. If there is not, it may be determined that the hotter compartments do not need to be eliminated. However, if Micro-VCM is ever to be used for a customer to conduct industry standard testing, only compartments that can be kept within the temperature specs of ASTM E595 should be used. Other ways to heat of the copper bar can be explored as well, including manufacturing a new bar or trying to implement strip heaters instead of the current soldered resistor.

The desiccator used for keeping samples in before and after tests is currently a Tupperware container with two Hydrosorbent SG40 40 gram Silica Gel Canisters purchased from Amazon.com. These canisters are capable of reducing the relative humidity in the container to $\sim 1 - 2\%$. Once they become saturated, they can be baked in an oven to reactivate the silica gel so that the canisters can be reused. Two more silica gel canisters should be purchased so that the current canisters can be replaced when they become saturated, and then the canisters can be cycled through the desiccator. A different container can be explored for the desiccator other than the Tupperware container, such as a real glass desiccator that may be more successful in keeping water vapor out.

ASTM E595 mandates that sample materials be kept in a container at ~50% relative humidity and at ~23°C for 24 hours before conducting a test. Currently this is achieved by using two Humidpaks that regulate a Tupperware container to ~50% humidity, and by relying on room temperature to be roughly 23°C. This served as a decent stopgap measure for this thesis since time and money was a factor. In the future, more advanced options can be considered. Humidity and temperature-controlled ovens are available for purchase and are ideal for this application. However, these apparatuses are very expensive, one being quoted at ~\$7000. The next step down would be to design an airtight box with electrical feedthroughs that is equipped with temperature and humidity sensors, as well as devices to control the temperature and humidity. An Arduino could be employed for this application, but there are also cheap options for temperature and humidity controllers that should be explored that would be less time consuming and easier to implement.

The chamber is vented with nitrogen gas in order to disallow test sample to absorb water vapor from air while the sample compartment bar is cooled. Currently, the nitrogen is passed directly from the tank into the chamber. It was noted by Dr. Chad Immoos of the chemistry department that nitrogen gas can actually still be wet, which would mean test samples are still getting exposed to water vapor. ASTM E595 states that a Molecular Sieve 5A or equivalent filter should be implemented to filter the nitrogen gas through before letting it enter the chamber (the sieve is often used as a desiccant). Research should be conducted to see if this is necessary i.e. if our nitrogen is indeed wet, and if it is, a method should be developed to implement the filter.

The biggest obstacle to overcome to be able to conduct better testing with Micro-VCM is the need of a microbalance. As this thesis showed, error bars associated with TML and CVCM values that were calculated with measurements from the semi-microbalance are substantial. This

error is acceptable to a certain degree, and the semi-microbalance performed well in obtaining outgassing data for the validation tests conducted with the aluminized Kapton tape. However, for materials that experience less mass loss than the tape, the error associated with the balance will encompass the TML and CVCM values. Also, this thesis showed that the semi-microbalance made it very difficult to obtain meaningful data when it came to combined AO and outgassing testing. The effect of AO on outgassing could be very small (if it exists) and it is necessary to conduct testing with a balance capable of measuring down to the nearest μg for the effect to come out in the data. Not only would it be ideal to have access to a microbalance, but it would also be idea for the Aero department to have access to its own balance, whether that be a semi-microbalance or a full microbalance. Currently, the only access to a balance is through the chemistry department, which is not a good long-term solution for conducting outgas testing in the Space Environments Lab. Unfortunately, microbalances are extremely expensive, costing ~\$26000. Semi-microbalances are more affordable, but are still very expensive at ~\$7000. It will be very difficult to obtain either balance for the lab, but it should be a long-term goal.

There are multiple electrical changes that can be made to the Micro-VCM machine. As was previously mentioned, the entire machine was wired for a 3-phase to 2-phase power transformer, which the machine was plugged into at JPL. The wiring configuration enabled the machine to be self-contained, i.e. one power chord was able to power every component on the machine. Cal Poly's Space Environments Lab is equipped with 3-phase power, but a transformer similar to the one at JPL is ~\$1300. For this reason, components were broken up into different single phase circuits and the machine is no longer contained to one power cord. This can be remedied, however. The mechanical pump is now switched so that it no longer has to be plugged in and unplugged to turn on and off. Similarly, a 3-phase outlet should be added to the Micro-

VCM machine that mirrors those that currently reside on MAX and SCOTT; this outlet should be switched so that the compressor that runs the cryo pump on Micro-VCM can be plugged into it and then be turned on via a switch on the machine's front control panel.

The other main electrical augmentation that should be completed is to change the power source of the hoist motor. Currently, the original 2-phase plug that came with the machine powers the hoist motor. This means that the power cord must be plugged into the 3-phase outlet behind the Micro-VCM machine in order for the hoist to work. The same outlet must be used for the compressor, which means that a lot of plugging and unplugging takes place when using the compressor and the hoist. The hoist motor is a single phase motor with power requirements that are met by the standard 120 V, 20 A outlets in the lab, meaning that it should be able to be plugged into a single phase outlet in the lab. An attempt was made to reconfigure the switch that controls the hoist to be powered by a single-phase outlet instead of the 3-phase outlet. Although fundamentally the change should have worked, the hoist motor did not run off the single-phase power. There was not enough time to troubleshoot the issue, so the original configuration was restored. However, with more time, a solution can be found that will enable the hoist to run off single-phase power, which will eliminate the hassle of unplugging the hoist whenever the compressor needs to be used. Ideally, the entire machine would be reduced to one 3-phase power cord and one single-phase power cord. Both cords would remain plugged in at all times, and all components (hoist, mechanical pump, compressor, etc) would be turned on and off via switches on the front control panel. Great care should be taken when working with the electrical configuration of Micro-VCM. All components should be unplugged when work is being done, and extreme attention should be paid to any exposed wires that could inadvertently touch metal and charge a surface if the wires have been moved from their original spots. A deep

understanding should be had of how power is transferred through the machine's circuitry via the 3-phase power cord before any electrical work is conducted.

Besides changes and additions to be made to the Micro-VCM apparatus itself, there are some changes or investigations to be conducted on the procedures in running tests in the chamber. As mentioned in this thesis, the procedure used to test the aluminized Kapton tape differed slightly from the guidelines set by ASTM E595. The standard called for the tape to be adhered to aluminum foil and placed in an aluminum boat. The standard does not specify whether the sample should be rolled or cut in order to fit into the boat, however, the European standard does say that the sample should be cut into 10 mm strips in order to fit into the boat. The European standard mentions that films, too, should be cut and placed in boats. As was explained earlier, the aluminum foil and aluminum boats were not used in this thesis because they caused trouble during the validation process. However, part of the trouble was due to trying to match values to NASA's database, which has since been determined to be the wrong way to validate the machine. First, research should be conducted (again, JPL could be of use here to see what they do) to see how films are typically tested. The use of aluminum foil and boats should be revisited and implemented to see if test results using those procedures pass the 1/10 and 1/5 standard deviation tests. These methods can be adopted for testing tapes and films if tests are successful.

The final item to address for future work on Micro-VCM is further validation of the apparatus. The validation process was explained in detail earlier in this thesis. It was determined that it would likely be impossible to validate the apparatus through matching NASA's values, however, there could still be a way to gain insight into how the apparatus at Cal Poly is functioning compared to other test facilities. The European test standard states that the correct

way to validate outgassing test chambers is conduct an audit involving multiple test facilities. Seven test samples are selected and tested by each testing facility. The values are accepted if the TML/CVCM values of $> 0.20\%$ are within 20% of the average value of all participants in the audit, and if TML/CVCM values of $< 0.20\%$ are within $\pm 0.05\%$ of the average value of all participants in the audit. Other aspects of the audit include performing a blank test and ensuring that the collector discs do not experienced a mass variation exceeding $30\text{ }\mu\text{g}$, as well as inspections of the dimensions of the apparatus and associated equipment.

The inspections are not likely necessary, at least not in the near future; they would become necessary if Cal Poly was ever to conduct outgassing testing for a customer. The blank test is a very simple test for Cal Poly to perform, although it will be difficult to adhere to the $30\text{ }\mu\text{g}$ mass variation spec without access to a full microbalance. The test can still be conducted multiple time to gain a sense of what is happening when nothing is in the chamber and if contaminants are present. The most important aspect of the audit is the “round robin” style testing of the seven samples. While it is likely impossible that Cal Poly could ever partake in such an event with multiple industry standard test facilities, it may be possible to set something up with JPL where seven samples are chosen and tested at both facilities so that comparisons can be made. If JPL cannot conduct testing specifically in tandem with Cal Poly, it may be possible for Cal Poly to obtain samples of materials that JPL has already tested and make comparisons to their results. In this case, it would be ideal to get samples out of the same batch that JPL used in cutting their own samples so that accurate comparisons can be made. Tim O’Donnell could be a point of contact for this kind of operation.

8.2 MAX

MAX does not have nearly as many changes and improvements to be made on it as Micro-VCM; MAX has been a part of Cal Poly's Space Environments Lab for two years now and many of the kinks have been worked out.

One aspect of MAX that requires attention is its VUV lamp. MAX is equipped with a Hamamatsu VUV deuterium lamp that is capable of emitting VUV radiation between 115 – 200 nm. This spectrum is the spectrum that acts synergistically with AO in that it breaks bonds on surface molecules and thereby prepares atoms to be oxidized. In this way, MAX was designed not only to observe the effects of AO on materials, but also the combined effects of VUV and AO on materials. However, currently, the VUV lamp is not functioning properly. No effect was seen between VUV + AO samples and just AO samples the last time the lamp was used in testing. Also, VUV is known to cause discoloration in beta cloth. A beta cloth sample was placed directly underneath the VUV lamp for 24 hours, but no visual defects could be observed. The best way to determine if the VUV lamp is working is by purchasing a phototube that is capable of detecting VUV radiation within the range of 115 – 200 nm. The Hamamatsu R1187 Phototube is a product that accomplishes this feat, but it is very expensive. It may be required to send the lamp back to Hamamatsu for troubleshooting; multiple Hamamatsu techs have already been contacted on the phone and were not able to discern the problem. Max Glicklin is a great reference for troubleshooting the lamp as he made the original purchase.

Another part of MAX that can be changed for the better is the coaxial cable that brings RF power to the electrode within MAX. The cable in question is the one that is placed inside

MAX, not the one on the outside that is connected to the RF generator system. The coaxial cable is very long, and therefore goes through turns before getting to the electrode. The cable can be cut down so that the RF power will not have to travel as far to get to the electrode, which is more efficient and will help mitigate reflected power and arcing problems. The end of the cable that plugs into the electrode will have to be remade to be identical to its current configuration in order to be able to be plugged in to the electrode.

8.3 COMBINED AO & OUTGASSING TESTING

This thesis showed that a lot more work and testing needs to be conducted before being able to establish whether AO has any effect on the outgas properties of materials.

The first order of business would be to try and conduct the testing with a full microbalance instead of the semi-microbalance. The data taken in this thesis shows that a full microbalance is probably necessary to detect the fine changes that AO may have on TML and CVCM values. As mentioned earlier, however, it will be very difficult to obtain access to a microbalance, so more testing should be conducted with or without it.

More tests should be conducted on the aluminized Kapton tape and the ITO-Kapton-Al film since those were the materials that showed possible correlations between AO and TML/CVCM values. The aluminized Kapton tape showed a difference in its CVCM values, although the difference was not significant as it was bounded by the error bars. However, the difference was enough to warrant more testing, and the error bars were larger for this material because the typical repeatability value listed on the balance was used as the uncertainty instead of using the standard deviations found in the weighing process. The ITO-Kapton-Al tape was the most promising result from this thesis as far as showing a possible relationship between AO and TML. Retesting both materials multiple times via multiple procedures will illuminate whether

the results found in this thesis were outliers or if AO really does affect the outgassing properties of these materials.

Not only should the two aforementioned materials be retested, but new materials should be tested as well. It is possible that AO affects materials differently depending on the material properties. One material may respond to AO with completely different TML and CVCM values while another material shows no distinction. It will be very important to test multitudes of materials before arriving at a conclusion as to whether AO affects outgassing. Money and time restricted the number and type of materials that were tested for this thesis, but as time goes on, it should be possible to obtain new materials to test. Materials that are made up of multiple layers should be given special consideration, since one of the hypotheses of this thesis is that AO could erode away the top layers of a material to expose underlying layers that could then outgas more. The ITO-Kapton-Al film that was tested for this thesis contained layers of material and showed a possibility of being affected by AO.

Not only should more materials be tested, but more procedures should be considered as well. The most glaring procedural change that should be explored is the consecutive testing in the chamber. It was shown in this thesis that consecutive testing (i.e. testing without a break in between to bake out the chamber) may have resulted in contaminants from the chamber causing test samples to experience mass gain instead of mass loss. First, this phenomenon should be explored more, regardless of whether AO testing is present. It is important to know whether Micro-VCM must be baked out after every test, or whether perhaps it only needs to be baked out after the specific 60°C tests that were carried out as part of the procedures in this thesis. After exploring this phenomenon, changes can be made to Procedure 1 and 2 from this thesis so that test samples are placed into a desiccator for 24 hours after the AO testing so that Micro-VCM

has time to bake out. The procedure could be changed even further and samples could be placed in the 50% humidity container for 24 hours after AO testing. Samples could be measured for weight vapor regained values (WVR), which is part of ASTM E595, but was not a value considered for this thesis. WVR is the amount of water vapor that a material absorbs when placed in a humid container after undergoing an outgas test. The WVR for materials could be taken and then the samples could be outgassed again to see if AO affects the WVR and then the TML and CVCM after the materials have reabsorbed water vapor. It will be very interesting to see if there are any effects on TML and CVCM that were not found in this thesis after samples are kept in a desiccator or humidity controlled box.

Other procedural changes can be implemented as well. The AO exposure time can be increased or decreased from the 24-hour mark set in this thesis. Decreasing the AO test time could make it easier to test more materials in a shorter timeline. The preparation of materials can be changed as well. This thesis utilized long strips of material as test samples, but future testing could involve cutting 2 x 2 inch squared of material to place in MAX. This would take advantage of the test compartments that are inherent to MAX, instead of laying the material strips on top of the ground plate as was done for this thesis. The samples would have to be cut in order to fit into Micro-VCM, and not as many samples would be able to be tested at once, but conducting a series of tests over a greater time period should make this doable. Similarly to the Micro-VCM future work section, the Micro-VCM procedures can be changed so that the material samples that were exposed to AO can be cut, adhered to foil, placed in boats, etc. for Micro-VCM testing. If MAX's VUV lamp can be fixed as highlighted in MAX's future work section, the lamp can be implemented in testing to see if AO/VUV exposure has an effect on outgassing; the lamp can even be used in the absence of AO to see if test results shown in journal articles can be

reproduced that show a relationship between UV radiation and outgassing properties. There are most likely many changes that can be made to improve AO/outgassing test procedures, or if not to improve them to get a different take on what is happening and observe something different. Those who take over testing with MAX and Micro-VCM should not be deterred from deviating from this thesis and trying new things.

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APPENDICES

A. LESSONS LEARNED AND NOTES ON TEST APPARATUSES

A.1 MICRO-VCM

This thesis yielded a great deal of knowledge about the Micro-VCM chamber, especially since refurbishing the chamber was a large part of the thesis.

The first lesson learned deals with the right order to go about a project such as refurbishing the Micro-VCM chamber. The chamber came to Cal Poly in complete disarray from being worked on by JPL engineers and from being disassembled to be packed and shipped. The machine had not been run for at least five years. It follows that every aspect of the apparatus needed attention in order to get the machine back up and running and understand how the test apparatus worked. The process that was followed to begin work on the machine essentially worked by tackling smaller issues first before looking at big picture issues. For instance, the first task that was attempted and completed was getting the water-cooling unit up and running, which involved opening the electronics box to find and fix an open in the circuit and then learn how the unit worked. Other tasks that were taken on in the beginning were finding and cleaning/replacing each and every O-ring on the machine. These were tasks that needed to be completed at some point, but they were very specific.

A macroscopic approach may have proved to be more efficient in getting the chamber up and running. Instead of starting with smaller projects, the first order of business could have been trying to plug the chamber in and turning it on day 1. This would have exposed that the machine was not wired for our lab very early on in the process, which would have given much more time to fix the problem. Similarly, it would have revealed that the mechanical pump was not wired

correctly, and that the diffusion pump had to be replaced with a cryo pump, much earlier so that those things could have been taken care of. To be fair, there was a huge learning curve when it came to getting to know the system and learning about vacuum technology in general, and the smaller steps taken in the beginning assisted with the learning process. However, if another project of this magnitude was undertaken, the macroscopic approach would be considered.

Other than lessons learned about the process of refurbishing the vacuum chamber, Micro-VCM has many nuances about it that were discovered during this thesis work. One of these is the heating of the sample compartment bar. The sample compartment bar has a temperature gradient from the top to bottom where the top is hotter than the bottom, and the middle is hotter than both. However, this temperature gradient is exacerbated when the compartment bar is heated quickly. When a large voltage and current is applied to the bar from the variac power supply, the bar heats up very quickly, capable of reaching $\sim 125^{\circ}\text{C}$ in a matter of minutes. However, this also results in the temperature gradient becoming much more severe; the top and bottom of the bar can be separated by as much as $10 - 15^{\circ}\text{C}$ once one of them reaches $\sim 125^{\circ}\text{C}$. One would think that the bar would then equal out as it reaches steady state and the gradient would come back down to 2°C , but that is not the case. Once the bar is heated up and experiences a wide temperature gradient, it has proven to be impossible to obtain temperatures close to each other on the top and bottom. Therefore, it is important to heat the bar up slowly. The maximum voltage that should be set on the variac power supply is 25 V, not much more than the typical setting of 20 V to keep that bar at $\sim 125^{\circ}\text{C}$. 25 V will warm the heater bar slowly, meaning it will be a longer wait until the bar achieves 125°C , but it will warm the bar without causing a severe temperature gradient that causes most of the compartments on the bar to be out of spec.

Another strange aspect of the compartment bars is that the bars in Micro-VCM go to different temperatures. Micro-VCM is equipped with two compartment bars that straddle the cold plate to allow for double the number of samples to be tested. However, when one bar goes to 125°C, the other bar only reaches ~113°C. An amp clamp was used to determine that slightly less current flows through the resistor on the bar that reaches a lesser temperature. The lesser current could be the cause of the disparity in temperature, or it could be due to another cause. Regardless, currently only one bar is functional at a time, so both bars should not be used in tandem for testing.

It is very important to be able to measure the temperature of the compartment bars accurately. Micro-VCM is equipped with type-T thermocouples for this purpose. However, getting solid contact between the thermocouple and copper bar proved to be more difficult than expected. The first attempts to establish a connection between the thermocouple and bar involved using Kapton tape to adhere the thermocouple to the copper surface. This method failed, however. If any air gets trapped behind the Kapton tape, it puts the tape under immense pressure as the vacuum chamber pumps down, which results in the tape releasing from the copper and the thermocouple losing contact with the copper. Furthermore, the high temperature of the copper bar impedes the adhesive of the tape, which also results in the tape coming undone. To get around this problem, the thermocouples were placed underneath the copper cover plates on the top and bottom compartments of the compartment bar. The cover plates were screwed down very tightly to ensure solid contact between the thermocouples and copper surface. This method sacrificed two compartments (top and bottom) that could not be used for testing purposes, but it leaves nine compartments that are still useable. A third thermocouple is attached in the top-middle of the bar via a screw and washer.

An important part of preparing for an outgassing test is baking out the collector discs. ASTM E595 calls for the discs to be baked out at at least 125°C, but the standard does not specify that the bake out should occur under vacuum. ECSS-Q-70-02A, however, does state that the bake out should occur under a pressure of $<10^{-3}$ torr. Baking out the discs ensures that all materials are outgassed from the discs prior to testing so that the only mass change that the discs will undergo should be from collecting volatile condensable mass from the test samples. Baking out the discs under vacuum in Cal Poly's lab have shown better results than not baking out the discs. Therefore, tests should be conducted after preparing discs with a bake out under vacuum.

Another discovery that was made was that it is usually necessary to set the water-cooling unit at 23°C in order to keep the cold plate at 25°C. This makes sense because the cold plate is being subjected to radiation from the heated compartment bars, so water is going to need to be slightly cooler to keep the cold plate at temperature. The temperature of the water must be monitored to ensure that it is keeping the cold plate at 25°C; while the necessary temperature of the water is pretty consistent at 23°C, at times it needs to be tweaked. Also, the water-cooling unit should be filled with decalcified water so that calcium deposits don't build up in the unit, the tubes, or the copper plumbing on the plate. Aquafina water is decalcified and can be used for refills. The cooling apparatus of the water-cooling unit should always be completely submerged in water.

When conducting outgassing tests, it is often desired to conduct several tests in a row, as well as baking the chamber out at low pressures after a test to cleanse it of contaminants. In these situations, it is ideal to keep the compressor and cryo pump running instead of turning off the compressor and allowing the cryo to warm to room temperature. The cryo pump can be run for weeks on end before it needs to be regenerated by letting it warm to room temperatures. Keeping

the cryo cooled down allows for the vacuum chamber to be pumped down very quickly since time does not need to be allotted to allow the cryo to cool all the way down from room temperature to 10 – 20 K.

A huge lesson that was learned about the cryo pump was what compressors are able to run it. Micro-VCM is equipped with a Cryo-Torr 8 pump, as is SCOTT; MAX is equipped with a Cryo-Torr 10. The Cryo-Torr 8 can be run by a model SC compressor, 8600 compressor, or 1020R compressor. A CTI 8200 compressor was purchased to run the Cryo-Torr 8 on Micro-VCM, but it turns out that it is not powerful enough to run the cold head motor of the cryo pump. This is contrary to what CTI-Brooks claims about the 8200 compressor; indeed, CTI advertises the 8200 as being compatible with the Cryo-Torr 8. The only reason it was determined that the 8200 is not compatible was through talks with Dan Goebel. While the compressor was exchanged for a refund, this serves as a huge lesson to anyone working in the lab: whenever the lab is ready to make a big purchase (like a \$3000 compressor), there should be absolute certainty that the part is compatible with the lab. The instance with the 8200 compressor would have been hard to avoid, because even had CTI been called to check, they would have said that the compressor was compatible. For big purchases, it might be worth it to check with Dan or another source with experience to make sure the money is being spent on the right product.

Finally, all of the O-rings on Micro-VCM were found and either cleaned or replaced. This makes it unlikely that any O-rings will need to be replaced any time soon. However, if the need arises, many O-rings were purchased already. Before purchasing new O-rings, the lab's O-ring stock should be checked to see if the right size is already there.

A.2 MAX

MAX underwent some changes before being utilized for this thesis, and a few notes can be made about its functionality.

First, the MC2 controller is used to match the impedance in the system and eliminate reflected power. It accomplishes this by changing the tune and the load, which actually is changing the capacitance of variable capacitors within the box. Typical values for the tune and the load that achieved 0 watts of reflected power were ~38% for the tune and ~64% for the load. MC2 has the option to set the tune and load in automatic mode, which means that MC2 will automatically adjust the tune and load to get 0 watts reflected power. This method works relatively well, but at times during this thesis it was necessary to put the load in manual mode. If MC2 could not find a match point in auto mode, the load was put into manual mode and increased by several percentage points. Increasing the load by about 5 – 8% resulted in several more watts of reflected power. However, once this step was taken, the load was put back into auto mode. MC2 was then able to decrease the load automatically and find a match point that resulted in 0 watts reflected power. This method was used many times to eliminate reflected power from the system.

As was mentioned in the results section, MAX started exhibiting an arcing problem toward the end of testing for this thesis. First, the electrodes and ground straps were thoroughly cleaned, but this did not help the arcing problem. Next, more aluminum tape was placed around the coaxial cable, BNC feedthrough, and connection between the coaxial cable and electrode. This eliminated the arcing at first, but then it came back. Finally, all of the old aluminum tape was stripped of the feedthrough and cable and new tape was then put on. At first, this did not solve the problem, but then even more layers of tape were added and the arcing finally stopped.

This goes to show how important the aluminum tape is, and that it always takes more tape than expected for make the system functional. It also shows that after several months of use, the tape is bound to wear out; at that point it is necessary to replace the tape.

B. PROCEDURES

B.1 MAX SAMPLE PREPARATION

1. Where gloves whenever handling samples
2. Cut samples into 2 x 2 inch squares
3. Outgas all samples for a minimum of 48 hours below 200 mTorr
4. Remove samples from outgassing chamber and weigh them within 5 minutes
5. Place the samples onto ground plate between the eight screw holes
6. Cover the samples with the sample containment plate, using the arrow on the top of the ground plate and bottom of the containment plate for proper alignment
7. Secure the sample containment plate with the eight low profile ¼” – 20 screws, using the short screw in the forward most hole

B.2 MAX PUMP DOWN PROCEDURES

1. Ensure that all vacuum control panel toggles are switched to the off position
2. Make sure all service panels are closed and secure
3. Flip the 208 3Φ VAC breaker to the “on” position
4. Open the ball valve to the pressurized airline
5. Check the pressurized air regulator and ensure that it reads between 70-75 psi
6. Turn on the “Main Power” on the vacuum control panel
7. Turn on the Granville-Phillips 316 Vacuum Gauge Controller; convectron gauge 3 (CG3) indicates chamber pressure in Torr

8. Make sure all ports are closed, including the green nupro valve on the gas insertion line
9. Turn on the Mechanical Pump on the vacuum control panel
10. Turn on the Chamber Rough on the vacuum control panel; monitor the chamber pressure on the Granville-Phillips 316 Vacuum Gauge Controller

B.3 MAX OPERATION PROCEDURES

1. Pump down the chamber as described in Appendix B.2
2. Open the green nupro valve on the gas insertion line; use the needle valve to adjust the pressure in the chamber to 175 ± 10 mTorr.
3. Turn on the R301 generator
4. Set the power to 125 Watts
5. Turn on the MC2 controller
6. Adjust the load and tune capacitors to 50%; make sure the operational mode is in Automatic for both load and tune
7. Turn on the RF power using the switch on the R301

The capacitors on the MC2 should auto adjust and find a stable operational point where the reflected power is 0 watts. If there is still reflected power or if the capacitors motors begin to oscillate, turn off the system and refer to the MC2 manual.

8. Once a stable point has been achieved, adjust the phase and magnitude potentiometers on the left hand side of the AT3 until the output on the MC2 controller is 0 ± 25 mV
9. Maintain a pressure between 165-185 mTorr and record temperature values every hour

B.4 MAX SHUT DOWN PROCEDURES

1. Turn off the RF power switch on the R301 generator
2. Close the green nupro valve on the gas insertion line
3. Turn off the R301 and MC2 controllers
4. Let the samples sit in the chamber under vacuum until the average temperature on the ground plate is under 35°C

5. Close the Chamber Rough valve, turn off the Mechanical Pump, and engage the vent valve to vent the chamber
6. Remove the sample containment cover plate, and weigh the samples within 5 minutes

Caution: the DSS will be hotter than the ground plate, avoid contact with the DSS

7. Disassemble and store the apparatus as necessary

B.5 MICRO-VCM SAMPLE PREPARATION

1. Always wear lab gloves when handling samples and discs
2. Bake out appropriate number of collector discs at $>125^{\circ}\text{C}$ and $<10^{-3}$ torr for at least 16 hours; keep discs in desiccator for 24 hours after bake out
3. Cut samples according to ASTM E595 depending on what type of material is to be tested; samples should be between 100 – 300 mg
4. Add samples to aluminum boats and place them in humidity and temperature controlled box at $50 \pm 5\%$ relative humidity and $23 \pm 2^{\circ}\text{C}$ for at least 24 hours
5. Weigh the samples and collector discs with a balance having $\pm 1 \mu\text{g}$ sensitivity (if possible)
6. Place the collector discs on the cold plate of the apparatus, and the test samples in the sample compartments within the copper sample containment bar

Before placing the discs and samples into the apparatus, the cold plate and copper bar should be cleaned with a 50:50 mixture of acetone and ethanol

7. Screw the cover plates onto their respective compartments to block the entries, making sure not to tighten the screws too much so that the covers stay flush with the bar

B.6 MICRO-VCM PUMP DOWN PROCEDURES, CRYO PUMP OFF

1. Ensure all valves are shut (rough valve, black nupro valve, vent valve, etc.)
2. Turn on convectron gauge controller and cryo pump temperature monitor
3. Turn on Mechanical Pump
4. Open Chamber Rough valve
5. Rough out chamber to low pressure, i.e. 50 – 150 mTorr

6. Open Gate Valve
7. Allow pressure to pump down below 150 mTorr (50 mTorr ideal)
8. Turn on the Compressor
9. When cryo cools to 150 K, shut Chamber Rough valve
10. Turn off Mechanical Pump
11. Allow cryo temperature to drop to 10 – 20 K

Ensure the pressure does not rise above 150 mTorr during cryo cool-down

12. Turn on ion gauge controller once convectron gauge reaches ~ 1 – 6 mTorr and stops changing

B.7 MICRO-VCM PUMP DOWN PROCEDURES, CRYO PUMP ON

1. Ensure all valves are shut (rough valve, black nupro valve, vent valve, etc.)
2. Turn on convectron gauge controller and cryo pump temperatur monitor
3. Turn on Mechanical Pump
4. Open Chamber Rough valve
5. Rough out chamber to low pressure, i.e. 50 – 150 mTorr
6. Open Gate Valve
7. Close Chamber Rough valve
8. Turn off Mechanical Pump
9. Turn on ion gauge controller once convectron gauge reaches ~ 1 – 6 mTorr and stops changing

B.8 MICRO-VCM OPERATION PROCEDURES

1. Prepare and load samples as described in Appendix B.5
2. Close the Micro-VCM chamber and pump it down to $< 5 \times 10^{-5}$ torr as described in Appendix B.6 or B.7

3. While the chamber is pumping down, turn on the Brinkmann MGW Lauda RM 20 water-cooling unit and set at temperature (typically $\sim 23^{\circ}\text{C}$) to control the cold plate to $25 \pm 1^{\circ}\text{C}$
4. When the chamber has reached 5×10^{-5} torr, turn on the variac power supply to raise the containment bar temperature to $125 \pm 1^{\circ}\text{C}$ within 1 hour
5. Continue to monitor cold plate temperature and containment bar temperatures, making adjustments to the water-cooling unit and variac power supply if/when necessary
6. After 24 hours, shut down chamber as described in Appendix B.9

B.9 MICRO-VCM SHUT DOWN PROCEDURES

1. Turn off variac power supply
2. Turn off ion gauge controller
3. Close Gate Valve
4. Turn compressor off if cryo pump is to be allowed to warm up, otherwise keep compressor running
5. Set nitrogen pressure regulator to ~ 25 psi
6. Open black nupro valve to vent chamber with nitrogen; allow chamber to rise to 100 – 200 mTorr
7. Allow temperature of sample compartment bar to decrease to 50°C (or whatever temperature is manageable to retrieve samples)
8. Open vent valve and completely vent the chamber
9. Open chamber and remove samples from compartments using Teflon tipped forceps and remove collector discs from cold plate
10. Place samples/discs in desiccator
11. Turn off Brinkmann MGW Lauda RM 20 water-cooling unit
12. Weigh samples/discs within 30 minutes of removal, if possible