MODIFICATION OF A GROUND BASED ATOMIC OXYGEN SIMULATION
APPARATUS TO ACCOMMODATE THREE DIMENSIONAL SPECIMENS

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Atomic Oxygen Simulation Apparatus
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ABSTRACT

Modification of a Ground Based Atomic Oxygen Simulation Apparatus to Accommodate Three Dimensional Specimens

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The space environment presents various challenges when designing systems and selecting materials for applications beyond Earth's atmosphere. For mission success, these challenges must be considered. One of the detrimental aspects of the space environment is Atomic Oxygen, AO. Only present in harmful quantities in Lower Earth Orbit, LEO, AO causes significant damage to materials by breaking molecular bonds. California Polytechnic State University's, Cal Poly's, space environments laboratory features an apparatus capable of simulating this environment. Very thin or short samples were tested to observe the mass loss due to erosion of the sample material. Recent modifications to the system allow it to expose surfaces of three dimensional objects to AO rather than only those two dimensional objects. Simulating this effect on taller samples makes available the opportunity to test coupons that are then used in additional testing to measure the effect of that erosion on other properties. Challenges in adapting the AO system are explored and addressed, as well as some possible use cases for future work. As a use case, bending moment specimens were exposed to AO prior to testing in four point bending. Multiple regression models were constructed to determine variables contributing to slope changes between specimen pairs' linear-elastic regions of force-displacement graphs. Results show that AO exposed specimens had significantly gentler slopes in the linear elastic region of the force-displacement curve, meaning that AO exposure reduced structural rigidity of the coupons.

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Chapter 1

INTRODUCTION

In this chapter, components of the space environment and their interest to researchers will be explored. First, motivation and methods for studying the space environment are introduced. Afterwards, previous works for studying part of the space environment are discussed. Finally, this chapter will end with a look at how the capacitively coupled plasma, CCP, at California Polytechnic State University San Luis Obispo simulates this environment.

1.1 Why Study the Space Environment

The space environment presents a variety of unique challenges for spacecraft. These vehicles must operate in a setting differing dramatically from that of their designers. Neglecting the effects of any part of the space environment can hinder mission success at an alarming cost to stakeholders. A spacecraft's surroundings vary based on its orbit, yet some aspects are the same regardless of where the object is outside of Earth's atmosphere. As an object's orbital altitude increases, the amount and composition of the surrounding air changes. Lower earth orbit, LEO, can be defined as any orbit less than 2,000 km in altitude. Vehicles at these altitudes experience orbital velocities on the order of 7 km s^{-1} . Beyond LEO, Medium Earth Orbit, MEO, is designated to be orbits less than 36,000 km in altitude. A Geosynchronous Orbit, GEO, has an altitude of only 36,000 km. Vehicles at this altitude have an orbital speed of about 3 km s^{-1} . The change in air density at these altitudes both results in a vacuum as well as a lack of protection from harmful effects that are otherwise blocked from reaching Earth's surface. Additionally, the lack of atmosphere restricts the spacecraft to a

single mode of external heat transfer: radiation.

The environment is categorized by classes which are defined based on their composition, structure, or state of matter; the primary four being referred to as the neutral, plasma, radiation, and particulate environments[22]. The neutral environment is essentially an extension of Earth's atmosphere, and therefore is primarily a concern for vehicles in LEO where neutral particles are most concentrated. Combined with orbital velocities, collisions with these particles can damage a spacecraft or alter its orbit and attitude via atmospheric drag. Atomic oxygen, AO, is a concern in LEO, especially between 180 and 675 km where it is the dominant species[33]. At these altitudes, diatomic oxygen, O_2 , becomes single oxygen atoms through a process called photo disassociation[33]. These oxygen atoms will collide with surfaces in the spacecraft's RAM direction, or the direction of the spacecraft's velocity vector. AO is corrosive due to its high collision energy, and its effectiveness is increased when coupled with vacuum ultraviolet radiation, VUV[19]. This is problematic for many spacecraft since polymeric materials, which are commonly used on spacecraft surfaces, are the most affected.

With a mean kinetic impact energy of 5 eV, collisions alone are often not energetic enough alone to remove these materials. They can, however, initiate chemical reactions, leaving a oxide layer on the surface[22]. This may (in the case of volatile oxides), result in mass loss. The quality describing how susceptible a material is to reacting with atomic oxygen is called the reaction efficiency or erosion yield and is measured in $cm^3/atom$ [13]. As different materials have different properties with some forming stable oxide layers and others volatile, one can protect an otherwise vulnerable surface by applying a protective coating. If a more vulnerable layer becomes exposed, however, it may be eroded out from under the protective layer through a process known as undercutting. Optical and thermal properties of eroded surfaces are altered due to AO-caused texturing and thinning. These are the result of

pits and cones that form around a stable material attempting to protect the materials underneath[19]. Engineers must account for AO, or else they risk mission failure due to unplanned changes in the surface properties of the spacecraft.

The particulate environment consists of natural and human made debris. The former being referred to as micrometeoroids, and the latter as orbital debris, together called MMOD. Collisions can be catastrophic depending on the size and speed of the particulate. Debris are created during collisions, launching of rocket bodies such as those that deliver payloads to Geostationary Transfer Orbit, GTO, or other such events. Natural particulates also exist, with smaller diameter particles less than 1 cm usually reaching speeds between 15-20 km/s[22]. These can be shielded against, and much larger items with diameters exceeding 10cm can be tracked and possibly avoided[19]. The greatest risk lies with the particulates larger than 1 cm in diameter but are still too small to be tracked.

The thermal environment presents a unique challenge for spacecraft due to the temperature extremes that can occur multiple times a day in brief cycles. In LEO, orbital periods are typically on the order of 90 minutes. For most of that time, the spacecraft is being heated by direct solar radiation as well as bond albedo. During eclipse, however, the main heat contributors are blocked and the spacecraft is in the Earth's shadow. The spacecraft that had to ensure that it would not exceed operational temperatures of its individual components while in sunlight now could freeze in eclipse. Less thermal control will be required for components that have wider operational temperature ranges. However, thermal fatigue of materials caused by the cyclical heat loads experienced in most LEO orbits can be a concern.

Figure 1.1: MISSE project specimens are placed onto trays and inserted into PECs[31].

1.2 On-orbit Testing

The Long Duration Exposure Facility, LDEF, was a spacecraft which was delivered and retrieved to and from LEO. Covered completely in experiments, its purpose was to study the space environment. Since then, the Materials Internal Space Station Experiment, MISSE, has continued that work by placing Passive Experiment Containers, PECs, on the International Space Station, ISS[19]. PECs, shown in figure 1.1, have held thousands of samples in order to observe how they hold up against the harsh environment of space. By attaching these in different locations and orientations on the ISS, different affects are analyzed. Some of these materials include components, coatings, and even biological materials [31]. These experiments contribute valuable information about the effect of the space environment in LEO on these materials. However, despite the success of these missions, collecting the data has been time consuming and expensive, making it difficult to qualify new materials for operation in space[19]. In the example of the LDEF, it was intended to only be in orbit for about a year, but instead was not retrieved until after over 5 years on orbit due to safety concerns at NASA.

1.3 Benefits and Limitations of Ground Based Tests

Ground tests are an alternative to flight testing which can reduce costs and lead time for testing materials. One frequently used standard to qualify materials for space flight is ASTM E595. The test involves heating a vacuum chamber to 125 °C at a certain pressure and observing the total mass loss, TML, of the material after 24 hours. There is also an option for measuring the collected volatile condensable material, CVCM. These two are of major importance as materials that out-gas go on to contaminate surfaces elsewhere on the spacecraft. This can have a detrimental effect on optical and thermal properties and can inhibit sensors. The thresholds for qualifying materials for space flight is 1% TML and 0.1% CVCM. The standard for evaluating materials with respect to AO is ASTM 2089. The benefit of these tests, of course, is that they are much less expensive, and much more practical to use, than launching new materials strictly for testing. There are, however, several disadvantages. In the case of the AO test, the exact conditions of the AO on orbit are often either difficult, impossible, or impractical to replicate exactly. The second major issue, and this is true for any space environment simulation, is that the total synergistic effects of multiple environments are not all repeatable.

There are several methods of simulating the AO environment. These include the use of plasma ashers, lasers, gridded or gridless ion sources, or microwave electron cyclotron resonance sources[19]. Plasma ashers typically use a capacitively or inductively coupled plasma, CCP or ICP. Systems such as these, which use radio frequency, RF, energy, tend to be the most practical for cost and simplicity while being scalable[19]. NASA Glen research center is home to one such system, as well as an electron cyclotron resonance, ECR, system. This operates by disassociating diatomic oxygen via collisions with electrons[19]. Ion sources typically operate on pure oxygen and discharge several species including AO, but are limited in flux capability. The

last method using lasers creates a blast wave using thermal heat[19].

ASTM 2089 is carried out by first dehydrating samples so that any mass loss observed is not due to dehydration during the period of exposure. The pressure during dehydration must be below 200 mTorr, and samples must remain under this pressure for 48 hours. Specimens thicker than 0.127 mm must be weighed periodically until mass loss is no longer measurable. Activities outside of vacuum, such as weighing samples, must be done quickly as to reduce the uncertainty associated with moisture uptake. The standard dictates a maximum time outside of vacuum of 5 minutes. Samples are exposed to AO for a period of 24 hours, after which they are weighed again within 5 minutes of removal from vacuum. Accompanying samples is a witness sample of a material for which its on-orbit erosion yield is known. The purpose of the witness sample is to measure the effective fluence, or arrival of AO per area, within the testing area.

CCP systems are able to produce AO, but it differs greatly from AO on-orbit. The ground-based system, however, is still able to generate material erosion similar to orbital AO by making up for low impact energies, such as 0.1 eV instead of 5 eV, with much higher densities. The result is an effective flux on the order of 10^{15} *atoms/cm²/s* or greater. Though surface roughing and changes in optical properties are still observed, the pits and cones described previously do not appear due to the lower, omnidirectional velocities in the CCP AO system. The omnidirectionality is a byproduct of the way that the AO is generated. The powered and grounded electrodes generate an alternating electric field within the vacuum chamber which contains air below 200 mTorr. The alternating electric field is strong enough to accelerate electrons, ionizing the gas. The ions are not sufficiently accelerated towards either electrode due to the alternating nature of the electric field. Electrons are still accelerated, however, causing more collisions and thereby turning diatomic oxygen into oxygen. It has been found that ambient nitrogen in the air is also ionized, but it

does not affect the results of the AO erosion[19].

1.4 Building on Previous Work

The goal of previous work completed at California Polytechnic State University San Luis Obispo, Cal Poly, in 2012 was to develop a ground based apparatus that could simulate the effects of the Low Earth Orbit Environment on materials through the reproduction of certain aspects of the neutral and radiation environment[19]. A vacuum chamber was retrofitted with a plasma generator and a deuterium lamp to study the destructive nature of atomic oxygen and vacuum ultraviolet radiation. The chamber, named MAX, has since been used in several studies to observe the effect of AO on different materials[21][12][18]. These included thin films, coatings, and small gels. Specimens were limited in size due to the diameter of the AO exposure area and the design of the plasma generator. The primary concern with thicker specimens was that they would interrupt the electrical path between two parallel electrodes which generate the AO.

The goal of this work is to modify the apparatus in order to allow thicker specimens to be tested in MAX. The motivation of this originated from the various applications for structural specimens. While most spacecraft structure design is driven by the launch environment, wherein materials have not typically been previously exposed to AO, there are still several on-orbit concerns for structural elements. These concerns aren't all related directly to AO, but exposure to it may affect structural responses to other environmental conditions.

Chapter 2

USE CASES FOR THE MODIFIED APPARATUS

In this chapter, use cases for the modified apparatus will be explored. First, various motives and possible applications for the chamber are introduced. Afterwards, sandwich structures are discussed. Finally, this chapter will end with a look at the study design.

2.1 Motivation for Research

Before making modifications to MAX, research was done to find possible use cases for the proposed system. This was necessary for several reasons, two of which were to justify the changes by showing that the new system would allow for beneficial research and to understand how the system should change or stay the same in order to accommodate a variety of possible testing coupons. Work regarding space environmental testing of materials abounds, with many of the effects of the environment being studied around the world. The initial question of interest was if there were valid applications of structural testing of AO exposed specimens. Information on the effect of atomic oxygen fluences on many different materials can be found, however most if not all of this information is in regard to thin films or coatings and not on bulk structural properties. As the design of most structural elements is driven by the launch environment prior to orbital insertion and subsequent exposure to the AO environment, there was some skepticism as to whether or not this work would prove beneficial.

2.2 Cases and Materials Considered

The first application investigated was the testing of debris shielding since MMOD is a structural concern on orbit. Hypervelocity impact testing has been completed on a variety of materials including but not limited to several metals, carbon fiber, and open-cell foam structures[37]. For the hypervelocity impact test, there is no testing standard, but in cases similar to this a projectile is accelerated to be on the order of projectile speeds in LEO. The study that was investigated achieved nominal projectile speeds of 6.8 km/s and observed the effects of changing materials and configurations but did not consider how these might be altered by the space environment other than MMOD. A proposed use case would be to mimic part of the study, but in addition compare the results of testing with and without AO exposure. Some concerns as to the feasibility of such a project at Cal Poly were brought up. The university is home to a rail gun which has accelerated particles beyond 1 km/s in the past, but it is not currently in operation.

An alternative to the hypervelocity impact test was to use a drop-weight impact setup. The magnitude of the speed, and therefore nature, of the impact would have been different. However, recent work at Cal Poly included the use of a Dynatup 8250 drop tower which could be used to test AO exposed specimens according to ASTM D7136[11]. Questions as to the comparability of this test to on-orbit impact were raised as other use cases were explored. In the face of these questions, another use case was selected. However, implications of the results of ASTM D7136 on hypervelocity impact behavior could be a focus of further research and work.

An abundance of publications about the effect of thermal cycling on carbon fiber parts were reviewed. One such work used a thermal vacuum chamber to cycle panels between -175 and 125 °C for 500, 1000, and 1500 cycles[36]. Coupons were cut for structural testing according to the appropriate standards for interlaminar

shear strength, flexure strength/modulus, longitudinal tensile strength/modulus, and longitudinal compressive strength/modulus. Another study with glass fiber material included cross-ply orientation of the fibers, but only exposed the samples to thermal cycling and not to vacuum conditions[3]. A primary interest in these studies was the effect that the thermal cycling had on the coefficient of thermal expansion, CTE, and several mechanical properties as a result of degradation of the polymer matrix. The final experimental work considered subjected graphite/epoxy composite materials to vacuum and thermal cycling but also included VUV radiation. Again, the main concerns were the change in thermoelastic and mechanical properties as a result of matrix loss[29]. Further exploration of these studies with the addition of the AO environment would provide new information. Only one experimental work was found that included AO in its study, but this merely reported the change in mass, tensile stiffness, and tensile strength of three samples, two of which were exposed to AO[27].

Unfortunately, many of the coupons used in testing standards to determine the structural properties mentioned previously were not especially thick. For example, the maximum recommended thickness for a tensile specimen is 2.5 mm[15]. Since one of the purposes of the use case was to gain understanding as how to operate the system with thicker samples, composite sandwich structures were considered. Sandwich structures, which will be further detailed in section 2.3, were the subject of numerical study which analyzed the thermally induced vibrations of a solar array in LEO[28]. However, this study only considered factors relating to the thermal environment and the panel material parameters but not any other effects like AO. The material is modeled with constant properties, which would not necessarily be the case for an actual solar panel on orbit. Results from a study observing the effect of AO on sandwich structures could inform as to whether changes in material properties due to AO would be beneficial to model. In any model, deflection of the solar panels would be desired to be small to avoid breaking any of the attached solar cells.

Figure 2.1: Diagram of a one dimensional material with length L .

The use case for the modified chamber was decided to be to observe the change, if any, in rigidity of a sandwich structure after AO exposure. The significance being that a change in the rigidity of said structure as part of a solar array would influence deflection behavior of these panels under thermoelastic stress in the linear-elastic regime. The concept can easily be explained by examining a one-dimensional material shown in figure 2.1.

The relationship between rigidity and deflection under thermoelastic stress can be seen by following the derivation starting with equation 2.1.

$$\epsilon = \epsilon^M + \epsilon^T \quad (2.1)$$

$$\epsilon = 0$$

$$\therefore -\epsilon^M = \epsilon^T \quad (2.2)$$

The material, with elastic modulus E and CTE α , is at a certain temperature, T , and length, L , before being constrained at each end. The total strain, ϵ , would be zero even if the temperature changed due to these constraints. This would imply that the mechanical strain, ϵ^M , and the thermal strain, ϵ^T , are equal and opposite. This is shown in equations 2.1 and 2.2.

$$\epsilon^T = \alpha \Delta T \quad (2.3)$$

$$\sigma = E \epsilon^M \quad (2.4)$$

$$\therefore \sigma = -E \alpha \Delta T \quad (2.5)$$

The thermal strain is simply the product of the CTE and change in temperature, ΔT , as shown in equation 2.3. The mechanical strain causes a stress, σ , in the material through the relationship known as Hook's Law shown in equation 2.4. Note that while the elastic modulus of the material is always positive, that the CTE can be negative. Most materials, such as metals, will have a positive CTE, meaning that they expand with an increase in temperature. Others, such as carbon fiber, will have a negative CTE, meaning they contract when heated. The negative CTE of carbon fibers allows laminate designers to achieve a near zero effective CTE by balancing the properties of the fiber with the matrix[29]. By equation 2.5 it can be seen that a change in temperature will cause either a compressive or tensile stress in the material depending on the sign of both the CTE and change in temperature. For example, if a material with a negative CTE got colder, the sign of the stress would be negative indicating that the material is being compressed by the constraints on each end as the material attempts to expand. This simplified example is enough to show that the rigidity of more complex structures is an important factor when considering bending behavior due to thermoelastic stress.

In order to have meaningful results, materials and fabrication practices would need to be as representative as possible of spacecraft solar panels. Carbon fiber face sheets with an aluminium honeycomb core were chosen due to their present use in spacecraft solar arrays and on-hand availability. Composite laminates are interesting to study because of their anisotropic properties. Many materials feature different elastic moduli under tension than they do under compression. Additionally, manufacturing

methods allow for great customization based on the intended application. One downside of using composite face sheets is how easily defects can be introduced into a part during the manufacturing process. An excellent overview of composites and manufacturing methods is provided in reference [11]. Aluminium honeycomb is often used due to its high strength to weight ratio. The core and face sheets would be bonded together using a film adhesive. This is common in industry, but a film adhesive was also crucial for the study design as will be explained in section 2.4. If the composition of a carbon fiber laminate was altered through interactions with atomic oxygen, the expected deflection behavior described in researched studies would be lacking an important factor.

2.3 Sandwich Structures

Sandwich panels are made up of a core material "sandwiched" between two face sheets as shown in figure 2.2. They have many applications, but are especially used in components that undergo bending. Weight-optimized sandwich design features a low-density core material with stiff high-strength face sheets. Usually foams or cellular structures are used as core materials. Honeycomb can be manufactured by essentially adhering layers of thin sheets with equally spaced lines of adhesive between them. Once the adhesive is set, the outer layers are pulled and the honeycomb repeated cellular structure results. The core typically has low mechanical properties, especially in comparison to the face sheets. The properties also vary by direction due to the manufacturing method. Across a continuous sheet, called the "ribbon" and noted with an "L", the properties are usually stronger than between sheets, called the transverse direction noted with a "W". This is shown in figure 2.3. The whole sandwich benefits from enhanced rigidity due to the distancing of the face sheets from the neutral bending axis - much like an I-beam. Sandwich structures do not

Figure 2.2: Simplified diagram of a sandwich panel concept with a honeycomb core bonded to face sheets using an adhesive[25].

experience some of issues that I-beams do under bending however, like torsion or buckling in the web. The former can be an issue because of the I-beam being an open cross section, and the latter can be an issue due to the instability of the web.

Several standard testing methods exist for testing face sheet, core, and assembled sandwich materials. Perhaps the most common tests for the assembled sandwich structures are the three and four-point bend tests. In either configuration, the sample rests on two supports, also called rollers or noses. The loading is applied by rollers at either one or two points. One point in the center is used for three point bending, and two points equally offset from the center are used for four point bending. These can be seen in figure 2.4. The deformation behavior and failure mode during the test will depend on the specimen geometry, material properties of each element in the sandwich structure, and the support/load spans. The analytic relationship between the force exerted and the crosshead deflection can be found for either case. This relationship for four point bending using variables from the schematic in figure 2.5 is shown in equation 2.6.

$$\delta = \frac{P(L - S)^2(L + 2S)}{48(EI)_{eq}} + \frac{P(L - S)}{4(AG)_{eq}} \quad (2.6)$$

The $(EI)_{eq}$ and $(AG)_{eq}$ terms are aggregates taking into account the cross sectional

Figure 2.3: Image showing how sheets of aluminium are bonded together and formed into honeycomb with directions L and W[5].

Figure 2.4: Diagrams of the loading configuration for three (Top) and four point bending (Middle and Bottom)[14].

Figure 2.5: Schematic of a sandwich beam for four-point bending[35].

geometries and moduli of the sandwich elements. An important note regarding equation 2.6 is that overhang is not a factor for crosshead deflection even though it is for predicting the failure mode. This is because equation 2.6 is a linear estimate where the core shear does not play a significant roll in the midspan dislocation. During failure, the displacement is no longer linear and equation 2.6 is not valid. Also notice that as the support span increases, so does the deflection at any given load, giving a decreased "observed" rigidity.

The aggregate bending stiffness, $(EI)_{eq}$, can be shown to be the summation of the stiffnesses of the face sheets and the core. Note that the centroidal axes of the face sheets are offset from the bending axis and require usage of the parallel axis theorem, resulting in a third term as shown in equation 2.7 where b is the sandwich width and t is the face sheet thickness.

$$(EI)_{eq} = \frac{E_c b c^3}{12} + \frac{E_f b t^3}{6} + \frac{E_f b t d^2}{2} \quad (2.7)$$

In optimal sandwich design, the third term dominates due to thin face sheets and a low modulus in the core. Note that this analysis assumes symmetric properties of the face sheets which would not be the case with carbon fiber face sheets featuring different tensile and compressive properties. One important takeaway from these equations is where the rigidity of the material could change with AO exposure. Degradation of the exposed surface could cause a thickness loss and/or change in elastic modulus from modified surface composition.

2.4 Study Design

Ubiquitous in structural testing is the force-displacement diagram. An example of a diagram generated directly from a mechanical testing machine is shown in figure 2.6.

Note that this is a raw output from the machine, and that prior to processing, the beginning term is typically offset to zero. Point A is located in what is called the toe region. In this region, the sample is not yet in full contact with all of the rollers due to the sample or loading noses not being perfectly parallel to the support rollers. Once the specimen is in full contact with all of the rollers, the linear region, containing B , begins. In this region, the force and displacement are directly proportional with some slope, m . At some unknown point near C , the curve starts to lose linearity but is still elastic, meaning that if the load were removed, the sample would return to its original position with no permanent deformation. Shortly after that, near D , the material yields and enters the plastic region. A peak load is experienced near E , until finally at F the specimen ruptures. There is a lot of valuable information that can be learned from the plot. However, for the purpose of this study, only the linear region containing point B will be observed. With the rigidity of the structure being the parameter of concern, analyzing changes in the slope of this curve in the linear elastic region are sufficient to make implications regarding the rigidity of solar array panels on orbit. There is no need to test to failure.

In order to determine if AO exposure caused samples to experience a reduction in rigidity, elimination of confounding variables in the experiment was of utmost importance. A paired study was designed in which bending moment specimens would be cut from a common panel. By doing so, effects from any variation in material, cure cycle, ply misalignment, and other conditions would be shared among samples from the same panel. In fact, this allowed otherwise inexcusable deviations from prescribed cure cycles to be performed without losing valuable data. Furthermore, pairs would consist of parallel adjacent samples, as those would be most likely to share manufacturing defects affecting rigidity. One member of a pair, the control sample, would be exposed to the vacuum environment while the treatment sample was exposed to AO and vacuum. This way, the effect due to vacuum conditions would

Figure 2.6: Raw force displacement curve of a sandwich specimen tested to failure output directly from the testing machine.

be shared, to some extent, by both samples. To achieve greater consistency, attention was given to the method used to bond the face sheets to the core. Using an epoxy structural adhesive that would need to be spread by hand was avoided. Inconsistencies in the spreading of the material were avoided by choosing a film adhesive which was more likely to evenly spread adhesive across the face sheets. Additionally, due to the deflection behavior being dependent on the support span as shown in equation 2.6, the support and loading spans would not be adjusted based on changes in the nominal lengths of specimens.

Many four and three point bend standards share similarities such as thickness to support span ratio of the samples. One of the major differences between four and three point bending setups can be seen in the resulting shear and bending moment diagrams for an isotropic beam shown in figure 2.7. In three point bending, the shear force within the beam experiences an instantaneous drop at the loading point. This is also where the bending moment is largest, and therefore the stress. In the case of four point bending, the drop in shear force is divided between the two loading noses,

Figure 2.7: Shear and bending moment diagrams for three/four point bending[32].

yielding a region of zero shear and constant bending moment. The value of having a middle section of constant maximum bending moment is that results obtained are less sensitive to stress concentrations created in the manufacturing process and are therefore more consistent. Under three point bending, the location of failure might be off-center due to a manufacturing defect at that location. The same defect, placed closer to the center of the load span, would fail at a lighter load due to the moment being greater closer to the center of the beam. In four point bending, the effect of this defect positioning is absent since the bending moment is constant between the loading noses. If the samples were tested to failure, the four point bending test would provide more consistent results and was therefore chosen over the three point bending test. Eventually, it was determined not to test the samples to failure, but the same standard was kept to since it still fulfilled the requirements of the use case.

The specific standard chosen was ASTM D6272-17 Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending. This standard was chosen because it

featured instructions for four point bending for laminated thermosetting materials[17]. Additionally, the geometry of the testing specimens, which must fit into MAX, were within the limits of the chamber unlike some standards for sandwich beams[16]. This, along with the chamber dimensions, determined the geometry of the samples as shown in table 2.1. The intent was to hold to the standard’s guidelines as much as possible while still producing sandwich beams that would be a fair representation of solar arrays on orbit. Included in the standard are specifications for specimen geometry and loading conditions. The smallest span-to-depth ratio recommended was 16:1. This was chosen since increasing this ratio would limit the thickness of the samples given the limited length of the exposure area. The width of the specimens and overhang were both a function of the support span, with the former being less than one fourth of the support span and the latter being at least 10% of the support span. After alignment of the loading and support fixtures, a rate of crosshead motion is calculated depending on the support-to-load span ratio. With no preference for either ratio, the ratio of 2:1 was chosen. The crosshead motion, R , was then determined by equation 2.8 provided by the standard.

$$R = 0.167ZL^2/d \quad (2.8)$$

where L is the support span in mm, d is the depth of the beam in mm, and Z is the rate of straining of the outer fibers in mm/mm. Per the standard, Z was equal to 0.01. The resulting crosshead motion was set to 4.25 mm/min.

Given the dimensions of the exposure area in MAX and the prescribed span to thickness ratio of 16 +/- 1, the sandwich thickness was aimed to be 1.12 cm. The total specimen length required for this resulted in not all of the specimen being directly in the field of view, FOV, of the AO. However, this was tolerated since almost all of the specimen would be in the FOV and this would be sufficient to observe changes in the

Table 2.1: Nominal dimensions conforming to specimen geometry of ASTM D6272 and the limits of the chamber

Property	Nominal Dimension
Length	20.0 cm
Width	3.18 cm
Core Height	0.64 cm
Total Height	1.12 cm

bending stiffness. It was determined to purchase 0.635 cm tall aluminium honeycomb and fill in the rest of the height with the face sheets. That thickness of aluminium honeycomb was readily available and was under the overall height limit of 1.12 cm. Though face sheets would usually not be this thick in most applications, it was desired to have as tall of samples as possible while keeping to the standards for the specimen geometry. That way, any issues encountered while exposing thicker samples to AO could be discovered, while obtaining meaningful results through testing standards.

This use case was simple enough to accomplish in addition to the other work involved in making changes to the chamber. Furthermore, it provided an essential experiment on how well the new system performed with larger specimens, while at the same time answering the question: does AO cause a detrimental effect on the stiffness of carbon-fiber sandwich panels with aluminium core, similar to those used on solar arrays?

Chapter 3

MODIFICATIONS TO THE ORIGINAL SYSTEM

In this chapter, the processes and motivations for modifying the chamber will be explored. First, the previously existing system is introduced. Afterwards, the design of the modified setup is discussed. Finally, this chapter will end with a look at the system replacement installation and testing.

3.1 The Existing System

The original setup was a capacitively coupled plasma, CCP, system created through the use of two parallel electrodes. One electrode powered, another grounded, the plasma is generated between them. The powered electrode lies above the grounded electrode, and is powered by a power supply system manufactured by Seren Industrial Power Systems, shown in figure 3.1. The package includes an RF generator, load matching network, and system controller. The generator is a Seren R301MKII that operates at a fixed frequency of 13.56 MHz and has a maximum power output of 300 W. In order to match the impedance load of the plasma generator, an AT3 matching network is paired with the generator. This impedance load was predetermined by an industry standard of 50Ω . This device is designed to protect the RF generator from internal damage while allowing the system to forward the maximum power to the plasma by eliminating any reflected signals produced in the load (cables, electrical connections, plasma). More details about the contents of these devices can be found in reference [19]. The system is controlled through user inputs and a Seren MC2 controller which automatically finds the matching impedance. A picture of the RF power system can be seen in figures 3.1 and 3.2[19].

Figure 3.1: Photograph of the Seren IPS R301MKII RF generator[38].

Figure 3.2: Photograph of the Seren IPS AT3 matching network and the MC2 controller[38].

The chamber, MAX, was constructed in one of Cal Polys high vacuum chambers. The chamber is a retrofitted Veeco Model 747 deposition chamber which has been modified for space simulation[19]. It consists of a pyrex cylinder approximately 50 cm in diameter and 32 cm tall. Initially, the chamber had two pumps: the first is a Welch Model 1397 mechanical pump with a pumping speed of 500 liters/min used as the roughing pump, and a cryopump. The roughing pump, which is still in use, can achieve a base pressure less than 10 mTorr. Updated procedures for operating the chamber can be found in appendix C.

The RF electrode was sized to consider: maximizing the AO flux generated, producing an exposure area large enough for material studies and analysis, and safety. The final choice was a 15.25 cm aluminium disc that is 0.9 cm thick. The material, a 6061 aluminium alloy, was selected due to its relatively high sputtering threshold which will reduce the amount of contamination that may occur. The electrode is mounted using four mounting holes for $\frac{1}{4}$ -20 alumina screws. These provide electrical and thermal isolation, while also assisting in alignment of the electrode with the ground plate. The power connector is inserted into a simple blind hole which is sized for a robust yet removable friction fit[19].

A dark space shield, DSS, surrounds the RF electrode. The DSS functions to minimize the secondary emissions from the electrode, thereby improving the concentration of the AO in the desired region. It does this by inserting a grounded conductive material inside the plasma sheath. The required gap distance between the electrode and DSS was found to be approximately 1.9 mm through empirical observations during preliminary apparatus testing. This gap distance was selected to eliminate any plasma generation between the electrode and the DSS[19].

The grounding plate is a 25.4 cm square aluminium plate mounting on an adjustable stand that allows for variation of the gap distance. A through hole in

the center of the plate is used to insert the operating gas, air, in between the ground plate and the RF electrode. In the original setup, an aluminium cover plate with a #8 mirror finish attached to the ground plate. This plate has four evenly spaced holes used for sample containment. These holes were precision machined to accurately and consistently control the witness and specimen samples exposure area. The holes are 2.540 ± 0.003 cm in diameter, outlining and sample exposure area of 5.06 ± 0.02 cm^2 . Eight low profile screws were evenly spaced around each sample area opening to apply even pressure and assure adequate masking of the samples[19].

The apparatus, modeled using computer aided design, CAD, can be seen in figures 3.3 and 3.4. The top half of the apparatus includes the RF electrode, the dark space shield, the RF coaxial power cable, ceramic spacers, and mounting hardware. The bottom half contains the ground plate, gas insertion line, and a cover plate for containing the samples. It also includes mounting hardware which is not shown. To ground the DSS and ground plate, 5.08 cm wide grounding straps made of type 101 ultra conductive copper 32 alloy were attached to each part respectively (not shown).

The final assembly of the original setup, with the cylindrical pyrex bell jar removed for clarity, can be seen in figure 3.5. A schematic of the chamber is shown in figure 3.6. Note in figure 3.5 that the hoist has been lowered in order to position the AO and VUV apparatuses in their actual test locations. Note also that the effects of VUV were beyond the scope of this project and the lamp was removed prior to the beginning of this work. Some minor differences between the pictured setup and the setup at the start of modifications existed. Those that needed to be addressed, such as two support rods for the DSS that extended down past the DSS, will be discussed, whereas less significant ones, such as the gas insertion line being to the left of the copper grounding strap, are ignored.

Designs for a replacement setup were required to still allow for the same testing

Figure 3.3: Cross Section of the Assembled AO apparatus[19].

Figure 3.4: Exploded view of the lower portion of the AO apparatus[19].

Figure 3.5: Photograph of the chamber with AO apparatus and VUV light source installed; bell jar not included[19].

Figure 3.6: MAX chamber vacuum schematic[12].

previously capable with the original four-hole plate, FHP. Additionally, the optimal design would allow a variety of specimen geometries and heights, would feature easy interchangeability, and would allow samples to be loaded and unloaded quickly to meet ASTM E2089's requirement that specimens remain out of vacuum for less than five minutes to avoid moisture uptake[13].

It was also preferable that the gap distance of 7.62 cm remain unchanged to avoid re-tuning the electronics. As part of the original setup's system calibration, the AT3 matching network had to be manually adjusted for testing. This process involved disassembling the matchbox in order to adjust the load and tune mechanisms. The details of this process are included in reference [19]. Great effort was taken to find a stable setup. A final configuration was found, which satisfied all of the operational constraints. This configuration disconnected the three fixed capacitors on the load mechanism while the inductor on the tune mechanism was only slightly compressed in the axial direction. The gap distance of 7.62 cm was one of few possible configurations and resulted in the highest AO flux without exceeding the maximum temperature limit[19].

In an effort to not have to repeat this work performed to find another optimal configuration, anything that would be known to require re-tuning the system was avoided. The two main limitations imposed were the gap distance mentioned above and the length of the coaxial cable which supplied the power to the top electrode. Altering either of these would change the impedance of the system, potentially beyond the controller's ability to match. Additionally, there were some concerns about altering the grounded electrode's area as this might adversely alter the size of the plasma sheath. The amount of area of the base plate would depend on the design for the specimens being tested in the chamber. Though this was not discovered to be an issue for the use case selected in this work, future base plate designers should be aware of this factor, especially if their design removes much of the material. There

Figure 3.7: An early concept of the changes to the chamber

could be a limit to the size of specimen that can be tested in the chamber that is smaller than the AO exposure area.

3.2 Modified Chamber Design

Initial designs for modification involved attaching the copper grounding strap to the cover plate and allowing what was previously the grounding plate, and now the base plate, to move up and down depending on the height of the sample being tested. The system would be equivalent to the original if the gap between the base and cover plates was closed for testing thin samples. The general approach was to maintain the grounded electrode at the same position while samples would be held beneath holes in that electrode. Also note that in this approach, the upper assembly was left as originally designed. Specimen geometries would dictate the shape and size of the holes on the cover plate which itself could be swapped out for testing of different specimens. Concepts were modeled in CREO Parametric 3.0[34] early on in order to visualize possible setups. One such early concept is shown in figure 3.7.

Shown in figure 3.7, this early configuration reduced the number of support rods (not shown) from four to the minimum number of points to define a plane, three. The base plate travels up and down the support rods and is secured in place with hardware. The base plate is still grounded, but it was unclear at this point if the hardware connections would form a sufficiently conductive path. One solution in the case that this became problematic was to have a sliding connection between the grounding strap and the base plate. The connection of the grounding strap to a stationary cover plate was necessary to allow flexibility in sample heights. The consequence was that either the cover plate needed a flange (shown in figure 3.7), the grounding strap needed to attach to the top of the cover plate where it could possibly interfere with the plasma, or the base plate would need a slot cut in it to accommodate the grounding strap in the case that a thin specimen was being tested.

These early designs presented several concerns. The most prevalent was the lack of protection for the sides of three dimensional samples. An object placed between the base and cover plates would likely experience side erosion as a result of the non-directional nature of the generated AO. This would be undesirable since AO is highly directional on orbit and it is believed that most use cases for the experimental apparatus would want to mimic that by restricting exposure to one surface. Another concern that arose was if the increase in grounded area would adversely affect the plasma sheath. Thirdly, it was unclear how to handle structural elements that have some uncertainty in their fabricated dimensions, as opposed the current system which constrained the exposure area. Finally, questions arose as to how to incorporate a witness sample if three dimensional specimens necessitated a gap between the cover and base plates.

Several of the above concerns were eliminated by the removal of the base plate, and instead using either casings made of shim stock or an aluminium tape mask to protect specimens from side erosion and/or to hold samples. A CAD model of this using a

plate with two slots cut in it for possible testing of rectangular elements is shown in figure 3.8 partially exploded. For future research, base plates may be machined to accommodate the structure being masked, but should note that the exposure area forms a circle about 15 cm in diameter. The height of the specimens should be no taller than 10 cm as to avoid extending up past the top surface of the base plate.

At the base of the figure are the supports that held the original configuration in place, with new through holes drilled in order to support the new system at three points. Three of the original four $\frac{1}{4}$ -20 threaded rods were used to hold the plate in place. The shim casings, represented by the brass-colored boxes at the top, are inserted into the slots in the two-slot plate to house rectangular specimens. The hope in using casings made of stainless steel shim stock was that the conductive walls near the specimen would function similar to the DSS and protect its sides from AO erosion. If after some testing this proved not to be the case, the last resort would be to mask individual samples. As will be discussed in chapter 6, it was discovered that masking was necessary. Masking was not preferred due to the reusability and ease of use of the casings. The final design of this specific plate, shown in figure 3.9, features a center rail between the slots where witness samples may be masked directly on the surface. This was chosen over adding a hole in the plate and applying a backing to make a witness sample. This decision was made mainly to simplify the machining process, but the process itself of masking witness samples was also suggested by an industry advisor. The uncertainty in the exposure area remains acceptable without the precision of the machined hole.

The aforementioned concern of how to mount witness samples was still an issue for the replacement for the FHP. With a single plate, there is nothing applying pressure to the samples to keep them pressed up against the underside of the plate. The solution to this was to machine pockets on the underside of the plate and backings to fill them and brace the samples. The backings are held in place by four #10-32 thumb

Figure 3.8: A later concept of the changes to the base plate.

Figure 3.9: Final design of the two-slot plate.

screws that hold four washers which support the backings directly. The underside of the final design is shown in figure 3.10.

3.3 Replacement System Installation and Testing

Two plates were machined from a 0.95 cm thick 6061 aluminium plate with a mirror finish using a computer numeric controlled, CNC, mill. The plate thicknesses were slightly larger than the original to allow more threads to be engaged in all of the threaded holes and to ensure sufficient thickness of material in each of the pockets of the FHP. The FHP had four 2.54 cm holes which were precision reamed to an accuracy of 2.540 ± 0.003 cm as were the holes in the original FHP. Two 206 by 45 mm slots were cut in the two-slot plate 25 mm apart. The center through hole for inserting the operating gas was 0.8 cm in diameter. The remaining two holes were tapped to accommodate #10-32 machine screws which secured the grounding strap to the underside of the plate. The center through hole, the three support taps, and the two taps for the grounding strap are all common features for any plate design using the new setup. The only variability is what geometry to cut for the specimens being tested.

Once the new plates had been machined and modifications to the supports made, the FHP was installed in order to test the new configuration. Immediately there were several issues. Two support rods for the DSS extended below the DSS and into the plasma, causing arcing during testing. The top assembly was partially disassembled so that the threaded rods could be cut to size. The length of the rods was shortened so that both the top and the bottom electrodes could be placed higher within the chamber while still avoiding extension of the rods beyond the DSS. Having the bottom electrode higher, in addition to having the support shafts to the bottom plate farther from the center, made a dramatic difference in the accessibility underneath the bottom

Figure 3.10: Final design of the replacement FHP.

Figure 3.11: Photograph of the chamber with the AO on and the new FHP installed. Notice the coaxial cable, covered in aluminium tape, extending down to the left of the DSS and into the plasma sheath only slightly.

plate. The increased convenience not only made changing out samples or plates easier, but also allows taller specimens to be tested without interfering with the supports below the plate. One unforeseen side effect of raising the DSS was that the coaxial power cable hung lower into the plasma than it did previously. The coaxial cable is covered with aluminium tape in order to prevent unresolvable reflected power. Having the cable deeper in the plasma sheath would require more frequent changing of the tape. However, changing the length of the cable would have opened up the possibility of having to re-tune the system. The solution to avoid these issues was to feed the cable farther around the DSS support rods before inserting it into the top electrode. The before and after pictures of this work can be seen in figures 3.11 and 3.12.

There was also an issue with the convectron gauge reading the chamber pressure. With the AO turned on, the gauge readings were erratic at best but were more often nonsensical. There were several causes theorized, one of which was that the gap distance was smaller than 7.62 cm and extra plasma was being generated as a result. Action was taken to ensure that the plate height was set to the correct gap

Figure 3.12: Photograph of the chamber with the AO on, and the two-slot plate installed. Notice that the coaxial cable is routed around one of the support rods for the DSS, preventing the cable from lowering further into the plasma after the height adjustments were made.

distance. Due to the nature of the hoist mechanism, it was difficult to find what height the bottom electrode needed to be. Various measurements to calculate the required distance from the supports to the plate were inaccurate. The solution to this was to tape a ruler to the DSS extending downwards to the grounded electrode to measure the gap with the chamber closed.

Another possible cause of the erratic convectron gauge readings was the new relative height of the air inlet. After the height change, the tube directing the operating gas did not reach the underside of the bottom electrode, possibly causing plasma to be generated in undesired locations in the chamber. A new 0.635 cm diameter pipe was cut, bent, and installed which arrived between the top and bottom sides of the grounded electrode.

Once the height was properly adjusted, the system ran without issues and was ready for its first tests using Kapton film as was common practice with the previous setup. Testing was successful, though it was noticed that the edges of circular exposure areas were not as defined as exposure areas observed from the previous configuration. This does not show up well in photographs, but the a sample from this test can be seen in figure 3.13. It was determined that there was interference between the machined pockets and the backings which held the samples in place. The interference prevented adequate pressure on the samples, and was resolved by sanding of both the pockets and backings, with emphasis on the areas away from the center.

In place of running more tests with the FHP to verify that fluence values did not unfavorably differ from the original setup, data from the use case experiments was used. This data is available in chapter 8. The resulting fluence values were similar to the previous apparatus.

Figure 3.13: Photograph of a Kapton sample after exposure in the FHP set on one of the plate backings.

Chapter 4

EXPERIMENTAL APPARATUS

In this chapter, the experimental apparatus used in this work, with exception of the AO chamber which was discussed previously, will be explored. First, the chambers used for dehydrating samples as part of ASTM-E2089 are introduced. Afterwards, the process of modifying and testing a desiccant chamber for dehydration is discussed. Finally, this chapter will end with a look at the systems for curing and bending the samples.

4.1 Dehydration Chambers

The space environments lab at Cal Poly is a shared working space. Graduate research, undergraduate lab courses, and industry work all require use of the facilities at varying times during the year. Two chambers commonly used for vacuum experiments are two identical chambers called Thing 1 and Thing 2 and are capable of reaching pressures on the order of 10^{-3} Torr. The chamber also features several feedthroughs for electrical connections and thermocouples that can be used to respectively heat the chamber and measure its temperature. Another chamber which is best suited for running ASTM E595 is the Environmental mass Loss Investigation chamber, ELI. The chamber is equipped for temperature control for measuring both TML and CVCM of materials. For the purposes of this work, these chambers were only used for their capabilities to store specimens under the 200 mTorr threshold for dehydration according to ASTM 2089 while issues with the desiccant chamber, Junior, were investigated. Pictures of these can be seen in figures 4.1 and 4.2 and more information regarding ELI can be found in reference [21]. Schematics of these chambers are also included in figures 4.3

and 4.4.

4.2 Desiccator

Due to the 48 hour dehydration process according to ASTM 2089, and in anticipation of increasing conflicts between the demands of graduate research and lab courses, a desiccant chamber was purchased. The desiccator, named Junior, is a Kartell model DYNCR 243065 large plastic desiccant chamber approximately 239 mm in diameter and has a clear lid. An 8 mm hose was clamped to its vacuum retention valve, and the other end was clamped to an assembly containing a KF 25 to 8 mm hose barb adapter and a KF 25 to KF 40 reducer nipple for attaching to pumps already used in the lab. A schematic of this chamber can be seen in figure 4.5. The hope was that Junior would serve as a dedicated chamber for dehydrating samples prior to testing in MAX. With a dedicated pump, tests could be run according to ASTM 2089 without interfering with other lab activities. Desiccant chambers are also known for holding vacuum well while disconnected from a pump. Previous studies have required mass measurements to be taken in a separate lab[21], and Junior would ideally address contamination concerns during transport. To allow Junior to be disconnected from a pump without losing vacuum, the hose was cut close to the hose barb adapter and the open ends were clamped to a ball valve. The ball valve was placed near the pump in an attempt to reduce the drooping effect of the weight of the valve on the hose. Since samples did not need to be transported under vacuum for this work, the vacuum retention capability of the desiccator was not tested. Instead, the chamber was continually connected to a pump.

It was discovered that daily AO tests could be consistently performed if the dehydrating vessel could contain up to 10 samples at a time. Stowing 10 samples in Junior at a time presented a challenge because each sample would need to be

Figure 4.1: Photograph of Thing 1, one of the student vacuum chambers.

Figure 4.2: Photograph of ELI used to dehydrate samples[21].

Figure 4.3: ELI chamber schematic[21].

Figure 4.4: Thing 1 and Thing 2 chamber schematic.

Figure 4.5: Junior chamber schematic.

Figure 4.6: Photograph of Junior with the accordion loop for holding samples with minimal contact.

mostly free of contact with any other surface that would inhibit outgassing. This was especially a challenge if the chamber had to be moved and specimens could shift. The first attempt was to bend a piece of shim stock into an accordion shape and have specimens rest inside of the troughs across the diameter of the chamber. This was able to fit eight specimens, but they were too much in contact with the accordion walls to be acceptable. The accordion was removed, rotated on its long edge, formed into a loop, and secured to the bottom of the desiccator. This is shown in figure 4.6. The troughs and peaks of the accordion around the loop formed ideal supports to hold specimens on their ends while having minimal contact with the specimens themselves. Additionally, the round lid assisted in preventing samples from adversely shifting during transport by coming close to the other ends of the sandwich beams which were sticking up. In this configuration, 7 samples were able to be stowed around the perimeter of the shim accordion loop, 2 were stored in the center, and 1 was stowed by resting it on top of the loop. This is shown in figure 4.7.

Due to the strict standards for dehydration, the chamber pressure for Junior

Figure 4.7: Photograph of Junior loaded with 10 samples

needed to be verified. The setup did not offer the capability to read pressure anywhere in the system besides the base of the hose where it connected to the pump. The low pressures on the order of 10s of mTorr observed near the pump caused the hose to collapse. A simple conductance experiment would reveal the pressure on the other end of the hose. In order to read pressure near the chamber, a temporary modification was made to the hose near the vacuum retention valve. It was cut in order to accommodate an assembly consisting of two hose barb to KF 25 adapters, two KF 25 to KF 40 reducer nipples, and a convectron gauge. The completed assembly can be seen in figure 4.8. Being able to read the pressure close to the entrance to the desiccator allowed a much better indication of chamber pressure than the single gauge near the pump. The initial test revealed that the pressure near the chamber did not go lower than 350 mTorr. Since the pressure in the actual chamber would only be as low as that reading, if not higher, action needed to be taken to ensure that samples would meet the 200 mTorr requirement set forth in ASTM 2089.

Known relationships exist between the conductance of an element and its geometry

Figure 4.8: Photograph of the test setup for the conductance test.

shown in equations 4.1 through 4.3[1].

$$C_v = \frac{3000\bar{P}D^4}{L} \quad (\text{viscous flow}) \quad (4.1)$$

$$C_m = \frac{80D^3}{L} \quad (\text{molecular flow}) \quad (4.2)$$

$$C_t = C_v + C_m \quad (\text{transition flow}) \quad (4.3)$$

where $\bar{P}$ is the average of the pressures on either end of a conductance element, D is the tube diameter, and L is the tube length.

In all flow regimes, the conductance is increased with a reduction in length. An increase in conductance would reduce the pressure difference between the chamber and the pump. The hose was cut to be about 60 cm, then the system was tested again. On the second test, it was noticed that the hose, though slightly compressed, had not collapsed completely. Additionally, the pressure near the chamber reached pressures below 150 mTorr with a pressure at the pump of 10 mTorr. Using a simple linear extrapolation, the estimated chamber pressure in Junior was 188 mTorr. Going

forward, Junior will be able to provide adequate vacuum to dehydrate samples.

4.3 Composite Curing Equipment

Composites that use resins often require heat and/or pressure to cure them. The heat is either supplied externally, or can be generated internally using a two part catalytic reaction[11]. Pressure needs to be applied to the part for two purposes. The first is to squeeze out excess resin, and the second is to ensure no air bubbles remain between layers in a laminate[11].

Both can be achieved using either a heat press or autoclave. The aerospace structures/composites lab at Cal Poly is home to an MTP-14 Compression/Lamination Press. The press uses hydraulics to force two parallel plates together while also heating them to desired temperatures. Within this same lab is an autoclave, which is essentially a pressurized hollow cylinder with spherical ends. The autoclave is capable of reaching pressures of 6.9 bar and temperatures of 538 °C while allowing vacuum connections to feed through. Both presses and autoclaves are commonly used in industry. They have their differences, yet either may be used in many cases. Parts in an autoclave are exposed to hot, often pressurized, air while being wrapped in bagging, films, and other materials. They are also often laid up onto a rigid tool. When using a hot press, the heat is distributed from the press to the part through the conductive tool. The pressure is applied to the part from the press and sometimes also an internal pressurized bladder that is deflated after curing has finished resulting in a hollow part. Presses have arguably less safety concerns since they don't involve a pressure vessel, but the conductive nature of the heat transfer can make autoclaves a more attractive option for having temperature control over the part.

4.4 Taking Measurements

Mass measurements were collected using a VeriTas S-Series precision balance. The readability and repeatability of which were respectively 0.001 g and 0.0005 g[12]. Force and position measurements were collected using a 5960 Series Dual Column Tabletop Instron mechanical testing machine. The apparatus is equipped with a 50 kN 2580 series static load cell and records data through an internal data acquisition system and then interfaces with a computer using Instron Bluehill software. The data is exported with CSV files with collected measurements for analysis. The crosshead and base adapter can be equipped with different fixtures to allow a variety of testing setups. The entire system, with wedge grips attached, is seen in figure 4.9. An example test that could be performed with this configuration would be a uniaxial tensile test, the most basic structural test where a rectangular or dog-bone specimen is pulled apart.

Figure 4.9: Photograph of the Instron mechanical test machine.

Chapter 5

SPECIMEN PREPARATION

In this chapter, the sample preparation process will be explored. First, the different materials used are introduced. Afterwards, the steps for fabrication are discussed. Finally, this chapter will end with a summary of the finished specimens and the differences between them.

5.1 Materials Used

Bending moment samples originated from three separate panels created in three separate batches. Each batch featured the same 0.64 cm tall DURA-CORE II 5052 aluminium honeycomb core, some properties of which are shown below in table 5.1. Honeycomb for vacuum applications is often vented to allow air to escape. This material, however, was not vented, yet was used since it was readily available. The first panel served as a test to see if this would cause any issues such as delamination of the face sheet while being put under vacuum. Since there did not appear to be any issues with this for the first panel, the material was used again for the second and third panels. It was unclear as to what the effect on outgassing would be due to substances being trapped within the honeycomb, but ASTM-2089 specifies instructions for verifying mass loss is minimal due to dehydration. The trial plate (and subsequent panels) didn't appear to be adversely affected.

What differed from each batch was a combination of the face sheet material, adhesive, and cure cycle. Each difference was based on what was available. An abundance of carbon fiber prepreg material was on hand, as well as some film adhesive. However, material specific to space applications was sought for. RS-36 M55JB 6K

Table 5.1: List of some known properties of the core material used to make the sandwich panels[2]

Property	Value @ 24 °C
Density	0.13 g/cm^3
Cell Size	0.64 cm
Compressive Strength	12.0 MPa
Shear Strength - L	5.1 MPa
Shear Modulus - L	770 MPa
Shear Strength - W	3.4 MPa
Shear Modulus - W	340 MPa

UD, a carbon fiber prepreg often used for solar arrays[9], was graciously donated by TenCate along with TC263 and RS-15H film adhesives. While these materials were being secured, on hand materials were used as a trial run. These materials included HexPly AS4/8552 carbon fiber unidirectional prepreg and LTA45ELNC film adhesive. Though properties of the latter were unknown, the properties of each prepreg and the adhesives from TenCate are shown in tables 5.2 and 5.3.

5.2 Face Sheet Fabrication

The fabrication process began by cutting out the plies of carbon fiber. The number of plies was determined by the desired geometry outlined in chapter 2, the height of the core, and the ply thickness. A 0-90 cross ply orientation pattern was chosen for simplicity. The outer layers of each skin was a 0 degree ply, and the layers alternated, each one perpendicular to the previous ply except in the case of the middle two plies in a mirrored configuration in which the number of plies is even. The balanced cross ply layup was chosen to avoid vulnerabilities which accompany purely unidirectional

Table 5.2: Datasheet information for carbon fiber prepreg materials used[8][24].

Property	HexPly AS4/8552	RS-36 M55JB 6K
Tensile Strength	2205 MPa	2041 MPa
Tensile Modulus	141 GPa	313 GPa
Compressive Strength	1530 MPa	993 MPa
Compressive Modulus	128 GPa	294 GPa
Shear Strength	128 MPa	75 MPa
Ply Thickness	0.11 mm	0.06mm
Outgassing	Not given	0.4% TML

Table 5.3: Some attributes of the film adhesives donated by TenCate. Note that the Lap Shear strength for the TC263 depends on if vacuum or external pressure was used[7][6].

Property	RS-15H	TC263
Lap Shear	21 MPa	19.6 MPa / 33.8 MPa
Cure Time	6 hours	2 hours
Cure Temperature	93 °C	121 °C
Outgassing	"Low"	0.34% TML

laminates.

Great care was taken in cutting each ply to get consistent sizes and square sides. In composites manufacturing, allowing any number of seemingly minor defects or overlooking small details during manufacturing can result in inconsistent or even poor part performance. For example, the symmetric design of the ply orientation is necessary to minimize residual stresses in the cured laminate. Plies were laid up in single pieces in order to eliminate the possibility of seams within the laminate where two plies in the same layer might overlap or leave a gap. This, coupled with the width

Figure 5.1: Tools used for cutting plies for the second and third batches.

of the prepreg material and tool size, limited the dimensions of the panel face sheets.

For the first batch, the autoclave in the Structures/Composites lab was being repaired so the hot press was used instead. The area of the metal plates limited the layup to a 21.5 cm square. Two plates were first covered with a protective film, then each layer of the prepreg was peeled and pressed on by hand until each laminate had 17 layers. They were then placed into the hot press and cured at 180 °C for 2 hours under negligible pressure. The ramp up and ramp down rates were respectively 3 °C and 4 °C.

While the first batch's face sheets were made in the hot press from the HexPly AS4/8552 material, the second and third batches' were made from the newly available RS-36 material using vacuum bag and autoclave processes. This material was preferred since it was known to be used for spacecraft solar arrays. No longer limited by the tooling dimensions for the press, the second and third batch laminates were 30.5 cm squares. That was the largest the laminates could be without splitting layers into multiple plies. Plies were stacked in a similar fashion as with the first batch,

Figure 5.2: Heat press used to cure composite materials in the Cal Poly aerospace composites lab[11].

Figure 5.3: Tool used to lay up carbon fiber face sheets

but the number of plies was increased to 30 per face sheet, 60 per panel, due to the thickness of the material.

The layup process used a typical vacuum bag procedure. A 61 cm by 91 cm aluminium tool was prepared with a nonporous release film. As layers were added to each laminate, they were periodically covered with another release film, breather cloth, and vacuum bag to evacuate excess air that could be trapped between the layers. This process, known as debulking, was repeated every 5 layers until all of the plies were stacked. Removing air pockets trapped between layers would lead to better consistency in specimen performance.

Due to the autoclave still not being available when the second batch was ready to be cured, another lab's large furnace was used. This furnace was capable of reaching 135 °C and pulling vacuum. This was not the prescribed cure temperature of 177 °C. A suggested adjustment to extend the cure time from 1.5 to 3 hours was made to compensate. This practice of increasing curing time at a lower temperature, though not ideal, is sometimes performed in cases such as these where equipment limitations

Figure 5.4: The oven in the Cal Poly mechanical composites lab used to cure the face sheets and adhesive for the second batch.

do not allow for strict adherence to a prescribed cure cycle. In this case, the deviation was acceptable due to the paired nature of the study. If the alteration to the cure cycle adversely affected the properties of the face sheets, the same effect would be shared between control and AO sample pairs.

The autoclave was available for the third batch, which only varied from the previous one in cure cycle. The prescribed cure temperature and time were followed, with inclusion of the optional external pressure of 4.1 bar. The change in cure cycle between the second and third batches caused a very noticeable difference in resin bleed-out. Shown in figure 5.6, the resin penetrated the release film and breather cloth in the third batch, something that did not happen with the second batch. This was likely caused by the combination of the elevated temperature and pressure. During heating, resins liquefy prior to hardening. With the external pressure squeezing it out of the part, less resin was retained by the laminate in the third batch than in the second. This was not tested for, but a reduction in resin content could result in loss of the sandwich strength and mass.

Figure 5.5: The face sheets for the second batch under vacuum prior to cure

5.3 Bonding of Face Sheets to the Aluminium Honeycomb Core

Face sheets were not abraded as is common practice to improve surface bonding to the core. The motivation being the desire to maximize similarities between sample pairs and to obtain consistency in observed results. It was feared that an inconsistent surface preparation would jeopardize that. Instead, each bonding surface was only wiped down with acetone to remove any dirt that had collected on the surface since curing. The bonding surface chosen was the flattest side of the face sheet. In the first batch, imperfections in the hot press tools showed up as divots on the surface. It was assumed that the flattest surface would provide a better bond than more heavily indented surfaces. Otherwise, no importance was placed on one side of a face sheet over another, rather effort was taken so that the directions of the outer layers were parallel. In the case of the second and third batches, the flattest surface was that which was in contact with the tool during the layup process.

The LTA45ELNC film adhesive was applied to the face sheets for the first batch.

Figure 5.6: The face sheets for the third batch upon completion of the cure cycle in the autoclave. Note the orange color on top of the plates from the resin being forced out of the prepreg.

This adhesive has no available information besides what is on the packaging from which we know that it expired in 2007 and is a low temperature film adhesive. Prior to adhering the face sheets to the core, the laminates were used as a stencil to cut the honeycomb core as shown in figure 5.8. Both face sheets were then pressed against the adhesive, which was then cut to the square shape of the laminates. After peeling the backing from the adhesive, the aluminium honeycomb core was "sandwiched" between the two face sheets and the entire assembly was placed again in the hot press at 90 °C for two hours with a slight pressure of 0.2 bar. Subsequent batches were bonded similarly but with a different adhesive with its accompanying cure cycle in a typical autoclave process. A panel immediately prior to being sandwiched can be seen in figure 5.9. The TC263 adhesive was used and cured in a vacuum bag process, shown in figure 5.7. The TC263 was chosen over the RS-15H due to the datasheet specifying its intended use for bonding honeycomb core to face sheets as well as its TML result of ASTM E595[7]. It had the added benefits of being a shorter cure time and listing a service temperature just over the maximum of what would be expected in an AO test. The second and third batches only varied slightly. The third batch had added the optional external autoclave pressure whereas the second only had vacuum. The second batch was also itself sandwiched between two metal plates within the vacuum bag. For the third batch, this was avoided since surface defects of the plates showed up on the panel's face sheets from the second batch.

5.4 Cutting the Panels into Beam Specimens

Once the adhesives were cured, the panel was ready to be cut into specimens. The samples were either cut using a tile saw in the Composites/Structures lab or the water jet at Cal Poly. The water jet was originally the preferred method for cutting the panels due to the accuracy of CNC machining techniques, its common use in

Figure 5.7: The second batch's panel curing the adhesive. Note that the position of the vacuum port on top of the specimen could cause undesirable bending were it not for the rigid metal tools sandwiching the panel.

Figure 5.8: Photograph of partway through the process of cutting out the aluminium honeycomb core material using a face sheet as a stencil and pressing a razor blade into the core.

Figure 5.9: Photograph of the adhesive applied to one face sheet, while the other face sheet has adhesive and the core material immediately before "sandwiching" the core.

industry for cutting fiber reinforced composites, and the reduction in the possibility of delamination which often occurs with many machining processes when cutting composites. Note that while moisture is a concern with uncured laminates, once a part is cured it may be cut with a water jet without fear of further moisture entering it. The two-dimensional specimen shapes are drawn and saved to a DXF file, shown in figure 5.10, which is then analyzed by the water jet's computer-aided manufacturing, CAM, software. It uses high pressure water mixed with a grainy abrasive material to cut two dimensional shapes through a material. While parts are cut, they are clamped at two locations on opposite borders of the part. The panel from the second batch can be seen clamped in the water jet in figure 5.11.

Several issues arose when attempting to cut specimens using the water jet. The first and most detrimental was not knowing how much material to leave between samples. The group that performs the service does not have experience cutting this kind of sandwich structure and initially suggested 2.5 mm. The second issue is the buffeting of the samples by the rebounding water after they are cut. The water as it

Figure 5.10: DXF file used to generate the tool path for the water jet for the second batch.

Figure 5.11: Photograph of the second batch's panel clamped in the water jet prior to cutting. The tape was placed so that the positions of the pieces could easily be tracked.

comes up disturbing any loose pieces can be seen in figure 5.12. The result is that already cut samples would bounce around and fall into the path of the water jet and get damaged. The third issue was the channeling of the jet of water through the cells of the honeycomb, resulting in ridges in the bottom face sheet. This can be seen in figure 5.13. The fourth issue was that the part would become unstable if the perimeter of the panel was cut. One concern with laminates in the water jet is that if the jet is initiated on the part, major delamination can occur depending on the material. For the first batch, there was no space on the panel to start the jet, so the cut had to initiate off of the part and move in, cutting through the border of the panel and destabilizing the part and causing further damage from the part moving unintentionally. This was not an issue with the second and third plates because there was an otherwise unused corner of the panel where cuts could be started. The fifth issue is the direction of the tool path. With the default settings, the CAM program had the jet trace the profile of a sample by first cutting the side far from the thin spacing created from cutting out the previous piece which is the side that would offer

Figure 5.12: Photograph of the water jet in use. Notice the violent behavior of the water coming up above the surface around the part.

the most support. The final support that a sample had was the flimsy 2.5 mm gap that would often snap off from vibrations during the cutting process.

Most problems were intended to be fixed with the second batch which featured an unused corner for starting the jet. However, some of the issues persisted, such as the gushing up of water causing samples to go under the jet. The result is that out of the first batch, only two of the three planned pairs of samples could be used for testing. Each sample featured a defect of some kind. Two samples were not straight, one had a jagged edge, and the other had an end of the piece that had a cut on it as shown in figure 5.16. The latter two were trimmed shorter than the others since one of them had a defect on one end and by cutting both of them, they remained the same length. Of the third pair that was to be cut from the panel, one sample was pierced through the center and was not usable. The other sample didn't get cut from the panel due to the lack of space left after the other samples had been cut out and the material had been jostled around. The second Batch produced five samples with no defects at all, but another two were completely disqualified so the panel produced only four out

Figure 5.13: Photograph side of a bending moment sample cut out with the water jet. Notice the ridges that match up with the walls of the honeycomb.

Figure 5.14: Photograph of the first panel's samples after being cut by the water jet.

of the five intended pairs. Endeavoring not to waste more time and valuable space rated material, the third batch was cut using a tile saw. Prior to cutting the panel into four point bending samples, it was covered top and bottom with tape to prevent delamination. Without the challenges of the water jet, the third panel produced five out of the five intended pairs with no observable delamination. Though improvements to the water jet process could potentially be made, reducing risk to samples by using a less complicated method for cutting the samples yielded better results. However, effort put forth to better the water jet process could prove beneficial in the future.

5.5 Summary of Batch Differences

A summary of the differences in preparation between the batches is shown in table 5.4. Prior to testing, it was unclear how much the defects, alternate cure cycles, and differences in materials would affect results. The first would lead to possibly significant differences in geometry between individual samples from any single panel while the latter two would lead to different material properties between panels. Due

Figure 5.15: Photograph of the samples cut by the water jet from the second batch. There was great improvement not cutting through the border.

Figure 5.16: Photograph of an example of an acceptable defect. Defects were allowed if they were believed to not affect the paired nature of the study (something like a puncture would be disqualified but a cut on the overhang was less significant for this study since there is no bending past the support noses).

to the paired nature of the study, the differences between the panels was not as much of a concern as the differences between samples within a given panel. Samples that didn't have any visible defects would form the most valid pairs and would produce better quality results.

Table 5.4: Summary of differences between the panels coming from the three batches.

Attribute	Batch One	Batch Two	Batch Three
Dimensions	21.5 cm square	30.5 cm square	30.5 cm square
Face Sheet	HexPly	RS-36	RS-36
Adhesive	LTA45ELNC	TC263	TC263
Cure Cycle	Hot Press	Modified Autoclave	Prescribed Autoclave
Cut With	Water Jet	Water Jet	Tile Saw
Samples	4	8	10

5.6 Finished Samples

Once the individual samples were cut, they were rinsed off, dried, and cleaned using acetone. Acetone is a solvent that easily cleans surfaces and evaporates quickly, so that little, if any, remains on the part when it is tested. Great effort was taken to keep track of the position and orientation of the samples as to preserve consistency in the results and to make sure sample pairs were not mixed. Treatment samples were masked all around their sides with aluminium tape with their specimen identifier etched into the tape in a specific location and orientation. The control samples were tagged with a small piece of aluminium tape and likewise labeled. The masking and tagging would potentially have an effect on the mass loss due to outgassing of the adhesive during the dehydration process, but this would not be detrimental as the standard dictates that the specimens be dehydrated until mass loss is not observed.

Figure 5.17: Photograph of samples after being labeled. Notice the treatment sample being masked around its four sides and the control only being tagged with a small piece.

A summary of all the samples, divided by batch is found in tables 5.5 through 5.7. It was decided after 22 samples had been made from the three panels not to continue making more. This was because results were already showing seemingly significant results and it did not appear that increasing the quantity of samples, though beneficial for the study, would be worth the cost of material and time investment.

Table 5.5: Summary of the 5 samples originating from first panel. Note that a 6th sample was not cut from this panel as was the original intent.

Identifier	Defect ^a	Group	Width/Length/Height (cm)	Mass ^b (g)
CT01	NS	Control	3.08/19.4/1.13	50.838
CT02	NS	AO	3.18/19.8/1.13	51.604
CT03	JE	AO	3.13/19.8/1.13	52.580
CT04	DQ	N/A	N/A	N/A
CT05	EC	Control	3.31/20.0/1.13	55.617

^a Defects are: NS (Not-Straight), JE (Jagged-Edges), DQ (Disqualified), and EC (End Cut).

^b Masses were measured without dehydration but after masking and labeling.

Table 5.6: Summary of the 8 samples originating from second panel.

Identifier	Defect ^a	Group	Width/Length/Height (cm)	Mass ^b (g)
CT06	DQ	N/A	N/A	N/A
CT07	NS	AO	3.19/18.9/1.06	46.579
CT08	NS	Control	3.13/18.9/1.06	46.608
CT09	DQ	N/A	N/A	N/A
CT10	NS, EC	Control	3.20/20.0/1.06	48.75
CT11	None	AO	3.14/19.8/1.07	48.074
CT12	None	AO	3.13/20.00/1.06	48.562
CT13	None	Control	3.14/19.8/1.07	47.908
CT14 ^c	None	Control	3.15/19.9/1.06	47.873
CT15 ^c	None	AO	3.15/20.0/1.03	47.695

^a Defects are: NS (Not-Straight), JE (Jagged-Edges), DQ (Disqualified), and EC (End Cut).

^b Masses were measured without dehydration but after masking and labeling.

^c Samples were aligned with the 90° direction, the rest with the 0° direction.

Table 5.7: Summary of the 10 samples originating from third panel.

Identifier	Defect ^a	Group	Width/Length/Height (cm)	Mass ^b (g)
CT16	None	AO	3.13/20.0/1.01	45.265
CT17	None	Control	3.11/20.0/1.01	45.170
CT18	None	Control	3.11/20.1/1.02	45.797
CT19	None	AO	3.13/20.1/1.03	46.241
CT20	None	Control	3.11/20.1/1.03	45.602
CT21	None	AO	3.13/20.1/1.03	46.501
CT22	None	Control	3.12/20.2/1.02	46.192
CT23	None	AO	3.14/20.2/1.01	46.508
CT24 ^c	None	Control	3.26/20.0/1.01	47.385
CT25 ^c	None	AO	3.20/20.0/1.02	46.236

^a Defects are: NS (Not-Straight), JE (Jagged-Edges), DQ (Disqualified), and EC (End Cut).

^b Masses were measured without dehydration but after masking and labeling.

^c Samples were aligned with the 90° direction, the rest with the 0° direction.

Chapter 6

EXPERIMENTAL TESTING

In this chapter, the steps performed for all of AO and bend testing will be explored. First, preliminary tests for side exposure and panel qualification are introduced. Afterwards, the steps performed for the AO testing are discussed. Finally, this chapter will end with the processes used for testing the beams in four point bending.

6.1 Preliminary Testing For Side Exposure and Panel Qualification

Prior to testing the bending moment samples, the question of whether or not the shim casings were sufficient to prevent AO exposure on specimen sides was investigated. If there was any doubt as to the shim casings' effectiveness, then the samples would need to be masked to assure restriction of exposure to the upward face. A simple experiment served as a preliminary test to see if any side erosion could easily be observed, visually or otherwise. A rectangular casing was formed out of stainless steel shim stock as shown in figure 6.1. A box made out of Kapton film and Kapton tape was made to fit inside the casing as shown in figure 6.2. The box had two pieces, a bottom piece with 5 sides, and a top piece that sat on top of the bottom piece with approximately 1 cm of overhang. The casing was fixed to the plate using aluminium tape, and an additional Kapton square sample was masked next to the Kapton box. It was later revealed that this positioning of the witness sample was outside of the exposure area and future witness samples were placed between the two slots. This was not discovered until after the manufacturing of the plate and has resulted in less area between the two slots than what would be desired if the plate was remade. The second slot was covered with aluminium tape in lieu of another sample.

Figure 6.1: Photograph of the materials used to shape and cut the stainless steel shim stock for the casings.

The top and bottom pieces of the Kapton box were separated so that they could have their masses measured separately, and therefore determine if the bottom piece experienced any side exposure. After performing the test according to ASTM 2089, both parts were weighed. The two pieces, shown in figure 6.3, both experienced mass loss, though visually the top piece experienced significantly more discoloration. After the results of this test, it was decided that further investigation into the side exposure concern would be avoided and that the samples would be masked with aluminium tape.

Prior to cutting of the trial sandwich panel with the Hexcel 8552 face sheets for testing in MAX, it was necessary to qualify it by doing a total mass loss, TML, test according to ASTM E595. The reason being that the outgassing properties of the prepreg and film adhesive were unknown and non-rated materials could have a detrimental effect on equipment in other chambers in the lab. The material was placed in Thing 1 at an elevated temperature for over 24 hours. The temperature was 117 ± 2 °C, short of the standard 125 °C, but was at the temperature for several

Figure 6.2: Photograph of the Kapton box placed in the two-slot plate using a shim casing. It is accompanied by a witness sample offset from the box.

Figure 6.3: Photograph of top and bottom pieces of the Kapton box after AO exposure. Notice that the bottom piece retained its normal color while the top piece has clearly been discolored.

more hours than specified. The pressure was as low as Thing 1 was capable, 7 ± 5 mTorr, instead of the prescribed 0.05 mTorr. The mass was measured using a balance accurate to the nearest 0.1 grams. The balance had to be used due to the weight of the entire panel exceeding the limits of the Veritas scale. As shown in figure 6.4 below, the plate had a mass of 411.9 ± 0.1 grams. After the test, the mass was measured to be just over 411.5 ± 0.1 grams which is about 0.1% mass loss. Even though the TML test was not performed to the exact specifications of the ASTM, it was believed that such a small mass loss at conditions experienced after 24 hours was sufficient to allow the material to be used in other vacuum chambers in the lab, including MAX. The deviations in temperature and pressure were permissible due to the exponentially decaying nature of material mass loss. Additionally, the purpose of the test was to ensure that the panel would not have an abundance of outgassing in MAX, which operates at a significantly higher pressure than what the panel was exposed to for the TML test. The other panels manufactured did not require this testing due to their known low outgassing properties.

Figure 6.4: Photograph of the balance reading of the trial panel before the TML test

6.2 Environmental Testing

Each treatment sample underwent procedures for ASTM 2089 (explained in chapter 1) while their control pairs remained under vacuum. Junior was initially used for both dehydrating treatment samples as well as keeping the control samples under vacuum. The desiccator could hold up to 10 bending moment specimens as well as several witness samples. After 48 hours, the samples were removed, weighed, and returned to the desiccator. This was due to the additional requirement for thicker specimens to continue dehydration until mass loss was no longer observed. After another 16 hours, the samples did not exhibit mass loss beyond the uncertainty of the scale, so the first two treatment samples were placed in MAX for the 24 hour exposure period.

While testing the first batch, it was noticed that the hose connecting Junior to Thing 2's pump collapsed. This caused doubts as to whether or not the desiccator had reached the required vacuum of 200 mTorr. The tray holding the samples for the second batch was moved to Thing 2 with its pump reattached. Eli was used

Figure 6.5: Photograph of MAX loaded with two bending moment samples and two Kapton witness samples.

to dehydrate samples for the third Batch. Thing 2 and Eli have convectron gauges allowing chamber pressure to be measured and controlled as to leave no doubt that the 200 mTorr ASTM specification was met or exceeded. Junior's desiccator pressure was later determined not meet the 200 mTorr requirement, but the effect was not believed to be detrimental because mass loss in the sandwich specimens was not as important as it is in other studies.

Each treatment sample was randomly assigned one of the two slots of the two-slot plate. Due to the height of the samples being comparable to the thickness of the plate and since the sample sides were masked, a convenient alternative to making two shim casings was chosen. Aluminium tape was placed on the bottom of the slots for the samples to rest on. The designated top side of the bending samples was chosen each time to face upwards and be exposed to the atomic oxygen. In addition to the two bending moment samples, rectangular Kapton witness samples were also placed near the center of the plate and masked with aluminium tape. One of these tests is shown in figure 6.5.

After the 24 hour exposure period, the AO was turned off and the treatment samples remained under vacuum until the time that they were tested in four point bending. This time varied anywhere from one to 12 hours and was based on the time it took the system to cool off sufficiently and the availability of the experimenter. The samples, both treatment and control, were removed from vacuum and weighed. The witness samples were also weighed to observe the effective fluence during each test.

6.3 Structural Testing

After weighing, samples were brought to the Structures/Composites Lab to be tested in the Instron machine. Control and treatment samples were tested in series by pair, but the order of which was tested first was assigned randomly. Each sample was tested twice, once with the top side facing up towards the loading noses, and once with the top side facing down towards the support noses. The order of which was assigned randomly in case the samples suffered from hysteresis. For the third batch of samples, however, the order of which side was faced upwards first was decided randomly for each pair rather than individual sample. All samples were tested within 10 minutes of leaving the vacuum chamber to avoid any effects of moisture uptake.

The testing was carried out according to ASTM D6272. The fixtures used comprised of two support rollers placed 165 mm apart and two loading rollers 83 mm apart. The setup, with a loaded sample, is shown in figure 6.6. The crosshead movement rate was set to 4.25 mm/min. The crosshead movement rate, support span, and load span was kept constant for all tests performed for consistency. Prior to testing samples from a batch, a dummy sample either from a disqualified sample or a spare piece from the panel was tested to failure. By observing the yield load, a load limit was established so that future samples would not exceed the linear elastic

Figure 6.6: Photograph of a loaded sample in the four point bending fixture.

limit. Each sample was loaded on the support rollers and centered by inspecting the amount of overhang on each side. After loading each sample, the loading rollers were manually lowered to connect with the sample. Connection was determined to be when the load measurement was positive and near 10 N. The test was then ran until loads near 1000 N were observed. At that point, the test was stopped and the loading noses were raised so the sample could be turned over and tested similarly. Samples did not display any damage after testing, indicating the the linear elastic region had not been exceeded in the loading process. A photograph of a loaded sample is shown in figure 6.6.

The exception was one case where the loading noses were commanded to return to the zero position which hadn't yet been set. Instead of being raised, the noses were lowered rapidly, destroying the sample before measurements could be taken with the specimen flipped over. The specimen in question will be discussed in chapter 8.

Chapter 7

ANALYSIS

In this chapter, the methods of analysis used in this work be explored. First, the parameters of interest are introduced. Afterwards, error analysis is discussed. Finally, this chapter will end with a look at multiple regression and how the calculated slopes can be normalized.

7.1 Parameters of Interest

Effective atomic oxygen fluence is determined by measuring the exposure area and mass loss in Kapton witness samples. The flux of oxygen atoms arriving at the surface of the grounded plate, be it the FHP or the two-slot plate, varies over the surface. However, measurements for individual or otherwise discrete witness samples are important for gauging the amount of atomic oxygen exposure during the test. Kapton HN is used as a witness material due to its known erosion yield on orbit of $3.00 \times 10^{-24} \text{ cm}^3/\text{atom}$ that has been found empirically. The effective fluence of the witness sample, F_k , can be determined by equation 7.1 shown below where ΔM_k , A_k , ρ_k , E_k are respectively mass change, exposed area, density, and on-orbit erosion yield all of the witness sample material [13].

$$F_k = \frac{\Delta M_k}{A_k \rho_k E_k} \quad (7.1)$$

The effect of the treatment on the stiffness of the sandwich beams can be observed in the change in slope of the linear elastic region of the force-displacement graph generated from collected force and position measurements during the four point bend test. An example of this was shown in figure 2.6. A one-sided paired t test was

initially considered with the following null and alternative hypotheses:

$$H_0 : \mu_d = 0$$

$$H_a : \mu_d < 0$$

where μ_d is the mean difference between the slopes of the treatment samples minus the mean slopes of the control samples.

The paired study, introduced in section 2.4, would compare specimens that had reasonably similar manufacturing defects, cure parameters, and environmental conditions which would otherwise affect the rigidity. However, it was later discovered that a multiple regression analysis was better suited. The reason for this is due to the nature of the slope calculation.

Curve fitting in general can be accomplished by using the following normal equation with n sets of observations and p coefficients:

$$A^T A \underline{\beta} = A^T \underline{y} \quad (7.2)$$

where $\underline{\beta}$ is a p by 1 vector of coefficients, A is an n by p matrix of the explanatory variables observed, and $\underline{y}$ is an n by 1 set of observations of the response variable. The simplest "curve" that can be fit is a straight line of the form $y = \beta_0 + \beta_1 x$, in which case equation 7.2 would be:

$$\begin{bmatrix} x_1 & x_2 & \dots & x_{n-1} & x_n \\ 1 & 1 & \dots & 1 & 1 \end{bmatrix} \begin{bmatrix} x_1 & 1 \\ x_2 & 1 \\ \vdots & \vdots \\ x_{n-1} & 1 \\ x_n & 1 \end{bmatrix} \begin{bmatrix} \beta_1 \\ \beta_0 \end{bmatrix} = \begin{bmatrix} x_1 & x_2 & \dots & x_{n-1} & x_n \\ 1 & 1 & \dots & 1 & 1 \end{bmatrix} \begin{bmatrix} y_1 \\ y_2 \\ \vdots \\ y_{n-1} \\ y_n \end{bmatrix}$$

Solving the above for the slope, β_1 , and y-intercept, β_0 , yields algebraic equations equivalent to the ones that have been developed to solve the linear regression problem[39]. These equations can be used on force-displacement data sets to fit the Instron data to a line. Using linear regression in this case is somewhat different than others in that in this case, the linear relationship is known. Instead of questioning if a linear relationship between variables exists, or "how linear" that relationship is, the question is what the coefficient that directly relates the change in displacement to a change in force is. Note that raw Instron data includes the crosshead position rather than a displacement. The y-intercept will reflect this by having a non-zero value. The slope, however, is unaffected by offsets such as these.

7.2 Error Analysis

To establish cause and effect between treatment assignment and stiffness, and to determine accurately the AO fluence during a test, all sources of errors should be considered and, where possible, accounted for. Errors that cannot be quantified, though many are taken care of by the paired nature of the study, may affect the validity of the results. For quantifiable errors, uncertainties are used. The uncertainty in any given measurement not only affects the worth of that measurement, but all parameters of interest which use those measurements in calculations. An error propagation equation for the resulting most probable error of any value q which is a function of variables $x \dots z$ is found below in equation 7.3 where each measurement uncertainty is preceded with a δ [39].

$$\delta q = \sqrt{\left(\frac{\partial q}{\partial x} \delta x\right)^2 + \dots + \left(\frac{\partial q}{\partial z} \delta z\right)^2} \quad (7.3)$$

In the case of the fluence calculation, this is fairly straightforward using differential calculus. Each term in equation 7.1 has uncertainties which can be easily determined

at the time of measurement. In the case of assumed values, an error on the order of the least significant figure can be used. The error propagation for equation 7.2 is less straightforward. However, this has been performed on its algebraic equivalents to yield the uncertainty in both the y-intercept and the slope. The equation for the latter is shown in equation 7.4[39].

$$\delta\beta_1 = \sigma_y \sqrt{\frac{\sum x^2}{\Delta}} \quad (7.4)$$

where $\Delta = n\sum x^2 - (\sum x)^2$

Note that the y measurement is assumed to be the only variable with error, σ_y , whereas the x variable is exact. Error in the slope when both measurements carry uncertainty can be computed by replacing this term for error in y to an equivalent term which includes error in x , σ_x [39]. Note that equations 7.4 and 7.5 make the assumption that the uncertainties are constant. This is true for the position measurements supplied by the Instron due to the final deflection being rather small[26]. The force measurement uncertainties, however, are proportional to the measurements themselves[26].

$$\sigma_y(equiv) = \sqrt{\sigma_y^2 + (\beta_1 \sigma_x)^2} \quad (7.5)$$

There are methods for incorporating variable uncertainty such as weighted least squares regression[39], however documented methods do not consider the case of one constant uncertainty for one variable, and a varying uncertainty for the other. Other less elegant techniques for slope error estimation exist, such as drawing error boxes at the first and final points and computing slopes using box corners. The decision was made to instead follow the procedure for calculating the error assuming a constant worse-case uncertainty in the force measurements.

7.3 Multiple Regression

The normal equation, equation 7.2, can be used for multiple regression without alteration. This technique was chosen over the paired t-test because the model is better aligned with the intent of the study and the nature of the data being analyzed. The intent of the study was to show that the coefficient that directly relates the change in displacement to a change in force is influenced by the AO treatment. To do this, the treatment category can be represented with one binary "dummy" variable, Q [10]. In this case, our variable would have a value of 1 if the recorded force and position observation was for a sample treated with AO, 0 otherwise. This means that multiple sets of Instron data are simultaneously considered. The addition of this variable adds not one but two coefficients to the equation the data is being fit to:

$$y = \beta_0 + \beta_1 x + \beta_2 Q + \beta_3 Qx \quad (7.6)$$

Note in equation 7.6 the interaction term, Qx . Fitting to this line using the normal equation will determine the best estimation for the interaction coefficient, β_3 , and for which a confidence interval may be determined. The null and alternative hypotheses are now revised to be the following:

$$H_0 : \beta_3 = 0$$

$$H_a : \beta_3 < 0$$

To make sense of this, take equation 7.6 and ignore for now the β_2 term since it will certainly be small. If the sample being tested has been treated with AO, the force predicted by the model, $\tilde{y}$, would be calculated by the following:

$$\tilde{y} = \beta_0 + \beta_1 x + \beta_3(1)x \quad (7.7)$$

$$\tilde{y} = \beta_0 + (\beta_1 + \beta_3)x \quad (7.8)$$

Knowing β_1 to be positive, the prediction will be higher or lower if the sign of β_3 is respectively positive or negative. If β_3 is zero, then the curve is the same regardless of AO treatment. The best fit for the line will be one which will better predict the force at a given deflection. Therefore, the alternative hypothesis is one sided since there is reason to believe that the treatment will reduce the slope. β_3 is essentially that reduction in slope since it will be subtracted from β_1 , reducing the effective coefficient multiplied by the displacement change to get a change in force. Note that this can be expanded. Indeed, coefficients and dummy variables may be added in order to observe the effect of other categorical variables such as specimen orientation relative to the laminate, order of testing to examine possible hysteresis, and orientation of the sample on the Instron rollers. Error analysis on this combined approach, far more complex than the initial linear regression technique, has not in any known study included experimental uncertainties. Instead, traditional statistical approaches are used[3].

After the model is created, it is validated using analysis of variance, ANOVA. This approach essentially compares the variance between groups with the variance within groups. ANOVA begins by calculating the regression sum of squares, SSR, by equation 7.9 where $\bar{y}$ is the mean value of the response variable. The regression mean square, MSR, is this divided by the degrees of freedom. The sum of squared errors, SSE, is calculated via equation 7.10. This is divided by the degrees of freedom for the error to get the mean square error, MSE. The degrees of freedom for the regression is simply the number of explanatory variables. For the error, it is the number of observations subtracted by the number of explanatory variables and 1. The test

statistic used comes from the F distribution for hypothesis testing. The equation for which is shown in equation 7.11[10].

$$SSR = \sum_{i=1}^n (\tilde{y}_i - \bar{y})^2 \quad (7.9)$$

$$SSE = \sum_{i=1}^n (y_i - \tilde{y}_i)^2 \quad (7.10)$$

$$F = \frac{MSR}{MSE} \quad (7.11)$$

The hypothesis test in this case is the following:

$$H_0 : \beta_1 = \beta_2 = \dots = 0$$

$$H_a : \text{At least one is different}$$

If the model is valid, partial t tests are then ran on each coefficient to determine significance of each term. The test of significance of these individual terms will determine if there is enough evidence to reject the null hypothesis that the AO treatment had no effect on the rigidity of the sandwich beams. Actual inference of the results of the statistical model will be discussed in chapter 9. The t statistic is calculated for each regression coefficient by dividing that coefficient by its standard error, s_{b_1} , defined in equation 7.12 where x_i and $\bar{x}$ are respectively the quantified explanatory variable and its average[10].

$$s_{b_1} = \frac{\sqrt{\frac{SSE}{n-2}}}{\sqrt{\sum_{i=1}^n (x_i - \bar{x})^2}} \quad (7.12)$$

7.4 Normalizing Results

After obtaining results, it was discovered that there could be some benefit to normalizing the slope measurements based on the specimen geometry. Though samples were all the same nominal size, small deviations would affect results. The testing standard used included an equation for the tangent modulus of elasticity in bending. Shown below in equation 7.13, this calculates the ratio, within the elastic limit, of stress to corresponding strain in MPa[17].

$$E_b = 0.17L^3m/bd^3 \tag{7.13}$$

where L , b , and d are respectively the specimen length, width, and depth all in mm. m in this case is the slope of the linear region of the force-displacement graph in N/mm.

Note that the bending modulus error must include the propagated errors from the geometry. Due to defects in the material due to the manufacturing process, this would be expected to be larger than the error for the slope only. Additionally, significance of the affect of the treatment on this parameter is not attainable using regression. Instead, ANOVA is still used as before but only with categorical variables.

Chapter 8

RESULTS

In this chapter, the results of the study will be explored. First, the results of the chamber validation are introduced. Afterwards, the results of the beam testing are discussed. Finally, this chapter will end with a look at the statistical results and if they indicate a cause and effect relationship.

8.1 AO Fluences with the Plate Replacement

The desired outcome of measuring fluences while testing the bending moment specimens was to verify that the modified system produced a similar effective AO flux. The duration of each AO test being approximately 24 hours, the expected fluence would be near 1.47×10^{21} atoms/cm²[19]. Equation 7.1 was used to compute the effective fluence and the associated errors shown in figure 8.1.

As shown in figure 8.1, the measured effective fluences from the two-slot plate were on the same order of magnitude as the original setup. The mean and median effective fluences were respectively 1.67×10^{21} atoms/cm² and 1.68×10^{21} atoms/cm². Though these are both greater than the original, they are still similar enough to be acceptable. Additionally, the original fluence is within the propagated error. Lastly, it should be noted that the witness samples tested on the two-slot plate were masked closer to the center of the AO flux distribution hitting the grounded plate. The distribution is most concentrated in the center, resulting in higher observed AO fluences.

One data point in particular worth discussing is the fourth from the left. This measurement was noticeably lower than all of the others, and had a significant amount of error, even considering that the scale of the y axis makes the lower half of the error

Figure 8.1: Results of fluence measurements while testing bending moment samples.

bar look longer. Though it's not certain, one possible reason for both of these is if the witness sample collected a small yet significant amount of adhesive from the aluminium tape used to mask it. The mass loss, and therefore calculated effective fluence, would be less due to the collection of the adhesive. The percent error would also be greater with the lower observed mass loss due to the uncertainty of the mass measurement remaining constant. The dimensions of the exposure area play a similar role in the relative errors. Even with this point being considered, it was determined that the chamber with the changes was generating acceptable AO fluxes over 24 hour tests.

8.2 Bending Moment Samples

The sandwich beams assigned the AO treatment had visual differences from their control pairs. As seen in figure 8.2, the AO samples appeared to have a more diffuse reflection of light whereas the control samples retained more of their specular, glossy, finish. The glossy finish is usually an indicator of a surface layer of resin, whereas

Figure 8.2: A treatment (left) and control (right) pair shortly after being removed from vacuum and tested in the Instron machine. Note the difference in optical properties as well as the residue that was readily wiped from the treatment sample.

a matte finish is more indicative of exposed fibers. Using a kimwipe, black residue was easily wiped off of the treatment samples. From these simple observations, it was speculated that the top layer of resin had been eroded and the fibers and resin beneath had begun the same process. However, it's possible that the change in optical properties could simply only be a result of texturing and thinning in the resin instead of exposed fibers[19]. The mean mass loss observed in these sandwich specimens after the 24 hour tests was 0.51% with a standard deviation of 0.06%.

Instron data was processed to eliminate data points in the toe region (region containing point "A" in figure 2.6). The slopes were measured and the bending modulus was calculated. The results for all paired measurements are shown in tables 8.1 through 8.3. Due to differences between manufacturing and materials (see chapter 5 for information about what differed between each batch), the tables are distinguished by which batch the pairs originated from.

Table 8.1: Testing results from the first panel (Hexply 2 hr @ 180 °C)

Pair #	Bending Orientation	m (kN/mm)		E_b (GPa)		F_k Avg. ($10^{21} \frac{atoms}{cm^2}$)
		AO	Control	AO	Control	
1	Up	2.27 ± 0.01	2.30 ± 0.01	65 ± 2	64 ± 2	1.3 ± 0.5
	Down	2.30 ± 0.01	2.29 ± 0.02	66 ± 2	64 ± 2	
2	Up	2.23 ± 0.01	2.46 ± 0.01	65 ± 2	70 ± 2	1.5 ± 0.5
	Down	2.30 ± 0.01	2.46 ± 0.01	67 ± 2	70 ± 2	

Table 8.2: Testing results from the second panel (RS-36 3 hr @ 135 °C)

Pair #	Bending Orientation	m (kN/mm)		E_b (GPa)		F_k Avg.
		AO	Control	AO	Control	
3	Up	2.85 ± 0.02	2.94 ± 0.02	86 ± 3	90 ± 3	1.8 ± 0.5
	Down	2.80 ± 0.03	2.92 ± 0.02	84 ± 3	90 ± 3	
4	Up	2.85 ± 0.02	2.94 ± 0.03	98 ± 3	105 ± 4	1.5 ± 0.5
	Down	2.89 ± 0.03	2.95 ± 0.02	99 ± 3	105 ± 3	
5	Up	2.84 ± 0.02	2.92 ± 0.02	103 ± 3	100 ± 3	1.5 ± 0.5
	Down	2.83 ± 0.02	2.90 ± 0.02	103 ± 3	100 ± 3	
6*	Up	3.64 ± 0.03	3.82 ± 0.03	144 ± 5	136 ± 5	1.8 ± 0.5
	Down	3.03 ± 0.02	3.48 ± 0.02	120 ± 4	124 ± 4	

* Pair samples were oriented perpendicular to other samples from the same panel.

Box plots showing the distribution of the percentage of slope loss in the AO treatment sample for each pair is shown divided by batch and pooled together in figure 8.3. The eighth pair, which was the one to show an increase in the slope of the AO treatment sample, is classified as an outlier in the combined distribution and not

Table 8.3: Testing results from the third panel (RS-36 1.5 hr @ 175 °C)

Pair #	Bending Orientation	m (kN/mm)		E_b (GPa)		F_k Avg.
		AO	Control	AO	Control	
7	Up	2.30 ± 0.01	NA	97 ± 3	NA	1.8 ± 0.5
	Down	2.50 ± 0.01	2.71 ± 0.02	105 ± 4	115 ± 4	
8	Up	2.72 ± 0.02	2.65 ± 0.02	110 ± 4	111 ± 4	1.8 ± 0.5
	Down	2.80 ± 0.02	2.53 ± 0.01	113 ± 4	106 ± 4	
9	Up	2.71 ± 0.02	2.76 ± 0.02	110 ± 4	112 ± 4	1.8 ± 0.5
	Down	2.71 ± 0.02	2.77 ± 0.02	110 ± 4	112 ± 4	
10	Up	2.72 ± 0.02	2.80 ± 0.02	118 ± 4	118 ± 4	1.8 ± 0.5
	Down	2.70 ± 0.02	2.80 ± 0.02	117 ± 4	118 ± 4	
11*	Up	3.01 ± 0.02	3.10 ± 0.02	120 ± 4	125 ± 4	1.8 ± 0.5
	Down	2.88 ± 0.02	3.06 ± 0.02	115 ± 4	124 ± 4	

* Pair samples were oriented perpendicular to other samples from the same panel.

when considering only data from the third batch. This point will be discussed shortly, but essentially the outlier has a speculated cause but cannot be removed completely from the analysis since the cause is not certain. Note that the box plots are generated using percentiles, the calculations for which can make it seem like data points exist where they do not. That is why there appears to be a second positive data point in the third batch.

Histograms showing the distribution between control and treatment slopes for batches 1 through 3 are shown respectively in figures 8.4 through 8.6. As seen in each batch, the average of the slope values were consistently lower in the AO treatment group. However, it is not possible to make inferences without the more robust analysis described in chapter 7. In figure 8.4, it can be seen that all of the AO samples had

Figure 8.3: Distribution of the percentage slope reduction in AO treatment samples.

slopes smaller than the smallest control sample. This was not the case with batches 2 and 3. It is believed that, since the first batch did not have many samples, there were too few data points to observe the variability between samples in the same treatment group. Variability between pairs needed to be accounted for. Additionally, other variables besides the treatment, such as hysteresis and testing conditions, could be factoring into the observed changes in slope. It can be seen from tables 8.1 through 8.3 that there are differences between AO treated samples and the control, even after propagating errors. However, determining causality would be of greater benefit, were it possible.

There was noticeable variability between sample slopes (beyond propagated errors) for control samples (which in theory should perform similarly) which brought up questions about the validity of the paired study. It is the experimenter's belief that this is primarily due to testing samples at different times (only four samples were tested in the Instron on any given day with the same atmospheric conditions). Additional concerns included the seventh pair which had an abnormally large observed

Figure 8.4: Histograms of the first batch's control and AO slopes (means in red).

Figure 8.5: Histograms of the second batch's control and AO slopes (means in red).

Figure 8.6: Histograms of the third batch’s control and AO slopes (means in red).

reduction in slope and the eighth which actually had an increase in the treatment’s slope. A few of these issues have probable causes. For example, the seventh pair’s treatment sample originated from a part of the panel close to the edge. It’s possible that it was cut too close to the edge where there is a higher chance of nonuniform ply stacking. The seventh pair also featured the control sample that was only tested in the ”down” orientation before being broken. The experimenter noticed that one of the loading noses after that test had displaced slightly, which could affect the results for the eighth pair since the load and support spans are a factor in the deflection behavior as seen in equation 2.6. Additionally, it should be noted that beams cut from a panel in different orientations are expected to have different results due to the non-isotropic properties of all of the materials involved (see tables 5.1 and 5.2). Finally, specimens did have small deviations from the nominal dimensions. This wasn’t thought to be an issue during the study design, but eventually this was compensated for by calculating the bending modulus according to the standard. Figures 8.7 and 8.8 show means for sets of samples that would be expected to perform similarly. For example, all of

Figure 8.7: Averages of the slopes for each batch.

the first batch's samples were oriented similarly, so they are all included. However, the third batch only had four samples that did not have any of the issues mentioned above. Note that the error bars on these graphs indicate the minimum and maximum observed respected slope or modulus. This was done since there were so few samples that it was deemed more appropriate than using the standard deviation.

One important thing to note is the difference in relative error between the bending modulus calculated value and the slope. As can be seen in tables 8.1 through 8.3, the calculated modulus also has more instances where the AO samples exceed their control pairs, though it should be noted that many of the differences are within error. Without more samples to get a better glimpse at the distribution of the modulus in either treatment group, ANOVA was used which included a factor for the pair, allowing more samples (like the perpendicular specimens) to be included in the statistical analysis.

Figure 8.8: Averages of the bending modulus data for each batches.

8.3 Paired Study Results

The ANOVA results, available in appendix F, showed that AO treatment was a significant factor in both the observed slope and the calculated bending modulus. The regression models for each batch were constructed after offsetting each force and displacement measurement by the first measurements for each sample. The purpose of this was to place the start of each line at the origin. Otherwise, the offset based on where the linear region started would affect the results. Terms with additional coefficients were added to equation 7.6 to account for hysteresis, sample orientation on the test fixture, and pair. The final addition being necessary as a blocking factor to account for the individual pairs not being related to the other pairs besides being from the same batch. The resulting F-statistic and corresponding p-value indicated that there was very strong evidence against the null hypothesis, or that not all of the coefficients $\beta_1, \beta_2, \dots$ are zero.

The next step in the paired study is to perform a partial t test for each coefficient.

In each case, the estimate for the treatment interaction was both negative and significant, meaning that the treatment caused a reduction in the slope of the force-displacement curve. The remaining interactive coefficients were interesting to examine although not the focus of the study. The hysteresis interaction was only found to be statistically significant in the first and second batches. Further investigation into this showed that only two of the eight samples from the second batch were tested first with their "up" side down. Bending orientation term for this batch was also statistically significant and was also positive like hysteresis interaction term. It is theorized that the random assignment of which orientation to first place the samples on the testing fixture simply did not split evenly enough with only eight samples. In other words, the "top-side-up" orientation of the panel was more rigid and a large proportion of samples from that batch were oriented that way first, giving the appearance that their second test was not as rigid because it was the second test while it was really because the sample was "top-side-down." Batch 1's assignment split evenly between the four samples, but with only two pairs, the observed hysteresis is more likely a result of the different defects that each sample had as shown in table 5.5. The significance of bending orientation term for each batch is not surprising as each face sheet will have different properties due to small inconsistencies in layering the plies.

The ANOVA results for the moduli, also available in appendix F, showed that the treatment also had a significant effect on the bending modulus. This is despite the increase in error and occurrences of stiffer pair treatment samples. Also significant was the pair factor. This factor captures several effects which include but are not limited to sample orientation relative to the original panel and atmospheric conditions during structural testing.

Chapter 9

CONCLUSION

In this chapter, the implications of the results be explored. First, the work accomplished and results are summarized. Afterwards, the possible future work modifying MAX is discussed. Finally, this chapter will end with a look at future work related to the use case.

9.1 Observed Effect of AO on Rigidity

The overarching question posed in chapter 2 was if atomic oxygen exposure was a factor that could change the thermoelastic behavior of sandwich structures on orbit. On-orbit experiments, though infinitely more valid than ground based ones, are costly and limited in availability. A compromise between validity, cost, and availability is made each time a component of the space environment is simulated in ground facilities. An effort must be made to maximize accuracy of environmental models, and therefore mission success. Ultimately, the space environment cannot be simulated with its entirety of synergistic effects using a ground-based apparatus. Designers must often rely on numerical models that they hope will be representative of the space environment. Strong evidence that a change in rigidity due to atomic oxygen is experienced by solar arrays on orbit made of carbon fiber sandwich panels or similar materials would necessitate consideration for accurate modeling and numerical analyses in lieu of flight testing.

Modifications were made to a ground based apparatus, MAX, in an attempt to find this evidence. The generated AO fluences during 24 hour tests were comparable with the original system, verifying that the modifications were not detrimental to

the apparatus' capability to simulate the environment. Sandwich specimens were fabricated using the same materials that have been used in solar arrays and were subjected to atomic oxygen. Afterward they were tested in four point bending, showing statistically significant reductions in the slope of the force-displacement curve and changes in the elastic modulus from their control pairs. Each pair of samples were compared individually, with some samples varying by as much as 10% in the slope.

Though the results of the study suggest that atomic oxygen can present structural concerns for spacecraft, they are only sufficient to warrant further investigation. Though much effort was spent in the design of the study, control and treatment samples still faced different conditions other than the atomic oxygen exposure which could have contributed to the observed results. When the AO is turned on, the power causes the system to heat up to approximately 93 °C. Additionally, the treatment and control pairs must be in separate chambers during the AO exposure, and they were stored at different pressures. In other words, it was verified that the assigned treatment resulted in a change in specimen stiffness, but it's possible that these results were skewed based on the limitations of the experiment.

9.2 Future Work on MAX

If divided into two parts, this work would consist of making modifications to MAX, and testing to see if there was a change in rigidity in bending samples when exposed to AO. There is plenty of work yet to do in both areas. The chamber itself is ready for testing materials with AO, but the system lacks the deuterium lamp that was included with the original system. The lamp was used to simulate the effects of VUV and to observe its synergistic effects with AO[19][12]. The needle valve that is used to bleed air into the system is challenging to turn in small enough increments to

get an equilibrium in pressure and could be replaced with a more easily controlled component. Junior accompanies MAX and would function best if it was setup with its own pump in order to be independent of schedules with the other chambers. Along with its pump, it could be placed on a cart so that the desiccator could easily be transported, if necessary, to the nearby lab which houses a micro balance scale for accurate mass loss measurements. The ideal setup would allow monitoring and changing in desiccator pressure to hold it near 175 mTorr, where AO samples are tested at, which would benefit future paired studies. ELI or another chamber would still need to be used to attempt to match the thermal loads experienced by the AO samples, however. Lastly, there was no measurable thickness loss in the specimens for this experiment. It would be beneficial for future studies to perform some image analysis on the exposed surfaces to see if any observations could be made about the thickness or composition of the eroded material.

9.3 Further Exploration and Refinement of the Use Case

Researchers also have the ability to expand on the scope of the use case. It was concluded in the paired study that there was a change in the slope of the force-displacement data collected by the Instron, but a more controlled experiment could attempt to quantify the amount of rigidity loss for a given effective fluence, especially if a method for propagating measurement errors through the multiple regression model was found. Additional controls, especially in the environmental conditions of paired samples, could be used to ensure that treatment effects were not due to the temperature rise when AO was turned on or the difference in chamber pressure between MAX and the chamber used for dehydration. Future study designs similar to this one could control the proportion of samples oriented in a certain way on the test fixture first as to eliminate confusion between sample orientation interaction

coefficients and those for hysteresis (β_3 and β_5 from this study, respectively). As mentioned in the previous section, samples could be tested with combined AO and VUV exposure. It should be noted though, that backings of solar arrays wouldn't likely be exposed to much VUV so it's possible that another spacecraft component would need to be considered in that study.

The multiple regression models for each batch of data being validated statistically, work could be performed in order to construct a model for simulation, building on previous numerical studies to predict the response of these materials on orbit. If a numerical model was built and could be validated by consistently predicting material properties after different lengths of exposure, the model would still have to be based on material properties since there is no guarantee that the model would be able to predict the response of a different material. The work involved in this would also then have to include pre-vacuum testing of materials since cured laminates will have properties differing from any spec sheets or each other due to the manufacturing process.

9.4 Adding an Environmental Focus

In addition to AO testing, as well as VUV if possible, another environmental effect that could be simulated is the thermal environment. A thermal vacuum, TVAC, chamber was recently donated to Cal Poly's Space Environments Lab. Though not yet fully equipped to allow cooling, the chamber will eventually allow thermal cycling of specimens. Though they unfortunately wouldn't be exposed to all three environments simultaneously, specimens could be exposed to AO, then mounted in the TVAC chamber and subjected to thermal cycles prior to structural testing. What would be better, were it possible, would be to be able to measure deflections of a cantilevered beam during the thermal cycling to observe the effects of the thermoelastic stresses,

mimicking thermal cycling of a solar array panel.

Already accustomed to testing coatings, films, and gels, MAX could be used to test these applied to the surface of structural members. Some coatings, such as silicon, are known to be effective at forming non-volatile oxides[19]. This effect has been observed previously, but faces problems as the oxides are more brittle and feature small irregularities that allow AO to penetrate deeper[21]. The result is that the surface layers may flake off. This could be especially problematic on a flexible member such as a long solar array which experiences bending due to thermoelastic stress. Protective coatings could also be intentionally damaged, simulating a manufacturing defect caused by (for example) a dropped tool, or some other possible scenario. The phenomenon known as undercutting, where material beneath a protective cover layer is eroded due to an exposed unprotected area[19], could be observed along with its effects on the structural integrity of the specimen.

9.5 Adding a Structural Focus

The analysis for this work did not necessitate the use of numerical solvers to predict the response of the material. Future work could endeavor to well define the material properties by measuring them prior to exposing coupons to the space environment. However, consideration must be given to the benefit of the study, as space-rated material is limited and many scenarios offer no real application for any results that would come of them. It would be interesting to vary parameters of the cure cycle and attempt to observe the difference in AO degradation due to things such as % resin content, cure temperature, cure pressure, preparation, and orientation of angle-ply laminates. Other structural testing methods, besides those already discussed in chapter 2, could include fatigue testing. Solar panels undergo thermal cycling, resulting in thermal fatigue. It's likely that this thermal fatigue is related to

mechanical fatigue, and it is possible that AO affects them both similarly, that is to say: detrimentally.

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APPENDICES

Appendix A

BASE TEMPLATE FOR FUTURE PLATE DESIGNS

The modifications to the lower half of the apparatus make plates easily interchangeable based on what sample geometries are being tested. It is essential, however, that three features are present on every plate in the correct locations. These are the positioning of the threaded holes for the support rods, the through hole for the insertion of air, and the two threaded holes for mounting the grounding strap. The dimensions for these are shown on the next page in figure A.1. Note that the plate design likely should ensure that there is space to mask at least one witness sample.

Figure A.1: CAD drawing with essential features for machinable plates.

Appendix B

OPERATING INSTRUCTIONS - JUNIOR

B.1 Prior to Operation

1. Ensure that the O-ring is sufficiently greased.
2. Secure samples inside the bowl.
3. Place the lid over the bowl.

B.2 Pumping Down Past 200 mTorr

1. Rest the vent plug and vacuum retention valve assembly over the hole in the lid.
2. Ensure that both the ball valve and venting plug are closed.
3. Turn the pump to the "on" position.
4. Open the ball valve.
5. Ensure minimal leaking. **Note** that these steps must be completed each time the desiccator is used to ensure that the chamber pressure reaches less than 200 mTorr without measuring it.
 - (a) Slightly lift the chamber by the lid to ensure that vacuum is reaching the desiccator.
 - (b) Wait several minutes as the pressure reading at the pump drops.
 - (c) Once the pressure is no longer dropping rapidly, push the vent plug in while twisting it until the pressure reaches a minimum.

- (d) Check all hose clamps to make sure that they are tight.
 - (e) While monitoring the pressure gauge, spray all hose interfaces with isopropyl alcohol. If a spike in pressure is observed, there is a leak and the hose clamp at that interface needs to be tightened more.
6. If the pressure near the pump does not reach 10 mTorr, there is still a leak in the system and it must be removed using the techniques enumerated above.

B.3 Bringing the Pressure Back Up to Atmospheric

1. Close the ball valve.
2. Turn the pump to the "off" position.
3. Twist and pull up on the vent plug until you start to hear the air entering the chamber. Avoid this getting too loud as venting too quickly can dry out parts of the chamber and cause cracking.
4. Once the pressure inside is in equilibrium with the atmosphere, the lid should lift off of the bowl easily. It is sometimes preferred to move the desiccator with the lid on. In this case, the vent plug and vacuum retention valve assembly can be lifted off.

Appendix C

OPERATING INSTRUCTIONS - MAX

C.1 Safety Concerns

1. The intent of this section is not to address all the safety concerns encountered in the Space Environments Lab at Cal Poly. It is the user's responsibility to ensure that all lab safety procedures are adhered to.
2. As with any space environments chamber, do not operate without first receiving proper training in safety and operation of this specific chamber.
 - Do not operate this apparatus alone. Those accompanying you must be instructed in how to shut down the system in the case that an accident occurs and you are unable to communicate.
 - You or someone working with you must also have Dr. Abercromby's direct number in case of urgent issues.
3. There is a bag of un-popped popcorn kernels mounted next to the radio frequency power source. The purpose of which is that in the unlikely event that potentially harmful amounts of energy are exiting the system, the popping kernels would serve as an indicator. If you smell, see, or hear the kernels popping, evacuate the lab and call Dr. Abercromby.
4. The system heats up to 93 °C during a 24 hour test. Heated elements include, but are not limited to, the coaxial cable, DSS, and the base assembly. It is recommended that users let the system cool under vacuum before removing samples for weighing. Another option would be to wear protective gloves when detaching the coaxial cable and removing samples.

C.2 Sample Preparation

1. Wear nitrile gloves to protect samples from contaminants on your hands. Fingerprints add weight.
2. Cut at least one kapton HN witness sample. The size of which will depend on the base plate being used. The FHP will accept up to four 5 cm by 5 cm squares placed inside the four pockets. Plates like the two-slot plate that don't have an opening for defining the exposure area will require dimensions dependent on where the witness sample will be masked. If placing the witness sample(s) near the center of the two-slot plate, the kapton should be cut to 3 cm by 2 cm unless otherwise desired.
3. Thin samples for studies on mass loss can be cut similarly to the witness sample(s) for the FHP. Structural elements should be prepared according to the standard(s) used in the study of interest and best practices depending on the material and what is available. Masking should be performed with aluminium tape around all surfaces except for those normal to the top electrode for which exposure is intended. This is due to the omnidirectional nature of the AO. Surfaces may be omitted from this process if it is known that erosion will not occur due to the surface's position and orientation.
4. Per ASTM 2089, samples (witness or otherwise) should be placed in a vacuum under 200 mTorr for 48 hours or longer as required until mass loss due to moisture evaporation is not measured.

C.3 AO Exposure

1. Ensure that all vacuum control panel toggles are switched to the "off" position.

2. Ensure that the main three-phase, 3Φ , power cable from the back of MAX is plugged into a 208 3Φ VAC breaker and rotated about 45 degrees clockwise.
3. Ensure that the 208 3Φ VAC breaker is flipped to the "on" position. If it is not, announce to the lab that you are turning on the 3Φ since it makes an alarmingly loud sound when toggled.
4. Ensure that the ball valve to the pressurized air line on the back of MAX is in the open position. There should be a pressure reading on the dial gauge in the back.
5. Flip the Main Power switch on the front control panel to the "on" position.
6. Turn on the Granville-Phillips 316 Vacuum Gauge Controller. Convectron gauge 2 indicates the pressure of the roughing line in Torr, and gauge 3 indicates the chamber pressure in Torr.
7. Prior to raising the hoist, ensure that the coaxial cable is not attached to the top of the chamber.
8. Use the hoist switch to raise the lid. **Note** that the hydraulic mechanism lags behind the switch. This poses a threat to grounding wires which are fixed to the lid, as they will break off if the hoist is raised too far. The hoist switch must be released in anticipation of this.
9. Lift the side lever that pushes against the bell jar and remove the glass.
10. Remove samples from vacuum. At this point, the five minute period has started.
11. Weigh the samples (witness or otherwise) and place them in the chamber.
 - For exposing thin samples in the FHP:

- (a) **Note** that prior to removing thin samples from vacuum, it would be beneficial to practice this process. The following are specific instructions on how to secure the specimens, but different steps can be used depending on user preference and ability.
- (b) Place samples in the center of the four backing pieces.
- (c) Place two backings, one at a time, inside adjacent pockets. Make sure that the sample does not move around so much that the exposure area intersects the edge of the material.
- (d) Hold these in place with one hand. With the other, secure them by screwing in a thumb screw and washer between the two pockets. Once the thumb screw is secured, the two backings may be released.
- (e) Place another backing inside one of the two remaining pockets and secure it by screwing in another thumb screw and washer between this backing and the already installed adjacent backing.
- (f) Repeat the above for the remaining backing piece.
- (g) Secure the last thumb screw and washer.
- (h) Ensure that all samples are secured flush against the underside of each pocket and that each backing isn't tilted.
- For exposing thicker samples in the two-slot plate or future plate designs:
 - (a) If using a shim casing to hold samples, fashion this out of acceptable chamber materials such as stainless steel. An alternative to making shim casings is to use aluminium tape to secure samples underneath the slots.
 - (b) The shim casing does not need to have overhang overlapping the top of the plate. Rather, it is recommended that the shim casing be taped to the interior of the slot, reducing damage to the top surface of the plate.

Aluminium tape should be sufficient to hold casings and samples in place.

(c) Place the sample inside of the shim casing.

(d) Mask the witness sample.

- **Note** that as much of the following as possible should be done in advance of removing samples from vacuum.
- Cut four pieces aluminium tape slightly longer than the four sides of the witness sample, and wide enough to be easily handled.
- Carefully fold the tape on itself along its length to create a straight flap that has adhesive on neither side. The flap should be narrow since large ones are less likely to maintain contact with the witness sample.
- After repeating the above with all four pieces, place three of them on the surface of the plate to form three sides of a square with appropriate dimensions so that the witness sample will slide underneath all three flaps.
- Place the final piece of tape to secure the witness sample.
- Flatten all flaps to ensure good contact with the witness sample.

12. Once samples are loaded, replace the glass bell jar

13. Lower the side lever that pushes against the bell jar.

14. Lower the lid using the hoist switch.

15. Attach the coaxial cable to the lid.

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

17. Flip the Mechanical Pump Power and the Mechanical Pump switches to the "on" position.
18. On the mechanical pump control box, press the green square "ON" button.
19. Flip the Chamber Rough Valve switch to the "on" position.
20. Monitor the roughing line and chamber pressures. Without inducing a leak, the system should pump down to 10s of mTorr.
21. The pump requires some time to warm up before stable equilibrium is possible. Allow the pump to run for 30 minutes.
22. Open the valve to the gas insertion line and adjust it to obtain an equilibrium pressure of 175 ± 10 mTorr. Monitor the chamber for several minutes to ensure stability.
23. Turn on the R301 generator.
24. Set the power to 125 Watts.
25. Turn on the MC2 controller.
26. Switch to manual adjustment mode and adjust the load and tune capacitors each to 50%.
27. Switch the adjustment mode back to auto for both tune and load. This is necessary or else the controller will not be able to reduce the reflected power.
28. Perform one last check for the chamber pressure. It should be steady at 175 ± 10 mTorr.
29. Turn on the RF power on the R301.

30. At this time, the MC2 will auto adjust to find a stable point where the reflected power, REF, is 1 or 0 Watts. If at any time REF is greater than 0 Watts, manually adjust tune and/or load until the REF is 0 Watts. If it is not possible to obtain 0 Watts REF, turn off the system and refer to the MC2 manual.
31. Adjust the phase and magnitude to be 0 +/- 25 mV each. These can be adjusted by turning the potentiometers on the left-hand side of the AT3 unit.
32. Maintain the system at these settings for 24 hours.
33. Turn off the RF power using the button on the R301 box.
34. Turn off the MC2 controller.
35. Turn off the R301 generator.
36. **Note** that the system is at an elevated temperature. Either wait for the system to cool or use protective gloves.
37. Disconnect the coaxial cable from the feedthrough port on the lid.
38. Close the valve to the gas insertion line.
39. Close the Chamber Rough Valve.
40. Flip the Mechanical Pump Power and Mechanical Pump switches to the "off" position.
41. Flip the Vent switch to the "on" position and fully open the valve to the gas insertion line. **Make sure** to flip the Vent switch to the "off" position when the chamber pressure reaches 700 Torr. Otherwise, the compressed air line will cause the lid to suddenly lift off of the chamber with a "pop."
42. Continue to vent the chamber through the gas insertion line.

43. Once the chamber pressure has reached equilibrium, use the hoist switch to raise the lid. While doing so, hold the coaxial cable out of the way in order to reduce strain on it. As before, **ensure** that the grounding cables are not strained by lifting the lid too high.
44. Remove the glass to gain access to the samples.
45. Remove the samples from the chamber to weigh them.
 - Using the FHP:
 - (a) Remove one of the thumb screws and washers.
 - (b) While holding onto one of the backings adjacent to where the screw was removed from, remove its other adjacent thumb screw and washer. The backing will then come away from the pocket.
 - (c) Repeat this process with one of the backings adjacent to the previous.
 - (d) For the last two, first support both backings with one hand while removing the screw and washer, then remove both backings with one hand on each.
 - Using the two-slot plate or similar plate:
 - (a) Remove whatever specimens were tested.
 - (b) Remove one side of the witness sample masking.
 - (c) Slide the sample out from under the remaining three flaps, making sure that the sample doesn't come in contact with any adhesive from the aluminium tape. If this is done carefully enough, three of the four aluminium pieces may be reused.
46. Weigh each sample (witness or otherwise) within 5 minutes from when the vacuum was lost.
47. Lower the lid when not in use.

C.4 Base Plate Replacement Procedure

1. Follow the instructions in section C.3 to open the chamber and remove the glass.
2. Prior to removing the plate currently installed, measure the height of the top surface of the base plate with respect to an easily reachable reference, such as the top surface of one of the support bars below the plate. The positioning of the height of the replacement plate will need to be the same.
3. Loosen a nut on each of the threaded support rods underneath the base plate.
4. With some freedom with the rods, they can be unscrewed from the underside of the base plate.
5. Using an allen key, unscrew the two #10 screws that attach the grounding strap to the underside of the base plate.
6. The plate should now be easily removed.
7. Place the new plate over the support rods, lining up the center hole with the opening of the insertion line.
8. Screw the support rods into the underside of the plate as well as the #10 screws that hold the grounding strap to it.
9. Place a level on top of the plate. Adjust the nuts on each support rod so that the top surface is the same distance from the reference as the previous plate and that the plate is level.
10. Once the plate is installed, continue with the steps in section C.3 to test that the setup can be stablized.

Appendix D

OPERATING INSTRUCTIONS - INSTRON

D.1 Safety Concerns

1. The intent of this section is not to address all the safety concerns encountered in the Aerospace Structures/Composites Lab at Cal Poly. It is the user's responsibility to ensure that all lab safety procedures are adhered to.
2. As with any testing apparatus, do not operate without first receiving proper training in safety and operation of this machine.
 - Do not operate this apparatus alone. Those accompanying you must be instructed in how to operate the system in the case that an accident occurs and you are unable to communicate.
 - Additionally, all activities in the lab require prior risk assessment paperwork to be completed in advance.
3. Never place digits or limbs between loading and support noses.
4. All persons in the lab must wear proper safety equipment while testing is being performed.

D.2 Four-Point Bend Testing

1. Turn on the tower and accompanying desktop machine.
2. Log in to the Bluehill application. The manual controls for the tower will then be enabled.

3. Move the crosshead up until there is sufficient space to install and adjust the fixtures.
4. Set the support span on the base fixture.
5. Install the base fixture but do not tighten.
6. Set the load span on the top fixture.
7. Install the top fixture but do not tighten.
8. Lower the crosshead so that the base rollers and loading rollers are nearly the same height.
9. Ensure that the rollers are all aligned properly while tightening them.
10. Raise the crosshead to allow a sample to be loaded.
11. Load sample on the support rollers.
12. On the computer, select the desired testing method.
13. Lower the crosshead so that it is near the sample.
14. Feather the crosshead down so that it contacts the sample while monitoring the load.
15. Stop lowering the crosshead once the load reads 10 N downward force.
16. If desired, zero the extension and balance the load. **Note** that it is recommended to set the zero extension above the sample so that return commands will lift the crosshead above the sample.
17. Click "Start" to begin the test.
18. The Instron will then run the selected testing method. However, the test may be stopped at any time by clicking "Stop."

19. If testing more samples, move the crosshead back up above the sample. You can do this manually, or by clicking "Return" if you set the zero extension above the sample.
20. Replace the sample with a new one and repeat the process of lowering the crosshead again until the load reaches 10 N downward force and clicking "Start" to run the tests.
21. Once finished testing samples, click "Save As" to choose a file name and location.
22. Click "Finish."
23. Navigate to the chosen location and transfer the output data to a flash drive or other device for use later.
24. Move the crosshead up until there is sufficient space to uninstall the fixtures.
25. Remove the top fixture.
26. Remove the bottom fixture.
27. Log out of the Bluehill software, shut the computer down, and turn off the tower.

Appendix E

OPERATING INSTRUCTIONS - AUTOCLAVE

E.1 Safety Concerns

1. The intent of this section is not to address all the safety concerns encountered in the Aerospace Structures/Composites Lab at Cal Poly. It is the user's responsibility to ensure that all lab safety procedures are adhered to.
2. As with any apparatus, do not operate without first receiving proper training in safety and operation of this machine.
 - Do not operate this apparatus alone. Those accompanying you must be instructed in how to operate the system in the case that an accident occurs and you are unable to communicate.
 - Additionally, all activities in the lab require prior risk assessment paperwork to be completed in advance.
3. Never attempt to open the autoclave door while it is under pressure.
4. When unlocking and opening the autoclave door, stand to the side of it rather than in front of it.
5. Check the air temperature before opening the autoclave. The system should be allowed to cool prior to retrieval of the part.
6. When removing objects from the autoclave, they can often be hotter than the air temperature reading. Use thick mittens to remove parts that could still be at elevated temperatures. Place these on a surface unaffected by heat. Note that cutting surfaces in the lab are warped by heat.

E.2 Curing Procedure

1. Parts going into the autoclave should already be bagged and ready for cure.
2. Turn the autoclave power on by turning the large lever on the control box on the left side of the autoclave. Note that sometimes a meaningless alarm will sound as the autoclave control program loads. This can be silenced using a large red button on the control box labeled "Silence Alarm."
3. View the perimeter of the door. It has teeth along the outside. If these are offset from the teeth on the door lock, then the door is open. If they are lined up, then it is locked. If locked, turn on the hydraulics on the right side of the autoclave by turning the rotary switch, then toggle the lever to rotate the lock. If you do not hear the hydraulics turn on, then the safety lever is likely engaged and must be released. If you hear a screech almost immediately, toggle the lever the other way until you hear another screech. Turn off the hydraulics once the door is unlocked. If it's already unlocked, then continue to the next step.
4. Connect a vacuum pump to the vacuum supply line to the autoclave and turn it on.
5. Attach a hose between the port to the vacuum bag and the vacuum feedthrough port inside the autoclave.
6. Set the part down and check the bag for leaks.
7. Shut the door.
8. Turn the rotary switch to activate the hydraulics for the door lock.
9. Toggle the lever to secure the lock.
10. Turn off the hydraulics and engage the safety lever.

11. Sign in to the autoclave controller software.
12. You may select a recipe which will automatically follow a prescribed cure cycle.
13. You may also control temperature and pressure manually. **Note** that the fan should always be running while the autoclave is in use. Also note that sometimes it is necessary to view the trend screen in order activate some changes when in manual mode.
14. Once the cure cycle has been completed, vent the pressure, if any remains, and allow the temperature to drop.
15. Return to the door lock mechanism while standing completely to the right of the autoclave. Disengage the safety lever and turn on the hydraulics.
16. Unlock the door using the lever, then turn off the hydraulics.
17. Push the door open and leave it open if the part needs to continue cooling.
18. Prior to removing the part from the autoclave, detach the vacuum hose and turn off the vacuum pump.
19. Remove the part.
20. Log out of the software and shut down the computer. Again you may need to silence an alarm while the software closes.
21. Once the computer is off, switch the power lever to the "off" position.

Appendix F

ANOVA OUTPUT

F.1 Regression Model

The coefficients accompanying the terms being considered are listed in table F.1 with a description of the term they are associated with. Note that the blocking factors have been omitted, and that the coefficient previously labeled as β_3 in the null and alternative hypotheses in section 7.3 is now β_7 .

Table F.1: List of included terms for curve fitting.

Coefficient	Description
β_7	AO Interaction
β_6	AO Treatment
β_5	Tested First Interaction
β_4	Tested First
β_3	Up Facing Interaction
β_2	Up Facing
β_1	Displacement

The results for each batch's partial t-tests are recorded below in tables F.2 through F.4. For the interest in this study, we are primarily concerned with the β_7 term. The p-values have been rounded to the nearest 0.01 due to the accepted level of significance of 0.05. Additionally, each partial t test for this term will be single sided due to the evidence that AO will reduce rigidity. The rest are double sided since there is no evidence that their associated terms will have either a positive or negative effect.

Some note should be made as to the level of significance used. This value is

Table F.2: Batch 1's partial t test results.

Coefficient	Estimate	Standard Error	t-statistic	p-value
β_7	-115.22	2.5935	-44.427	0
β_6	-4.5547	0.39177	-11.626	0
β_5	-11.225	2.5926	-4.3297	0
β_4	-8.2149	0.39177	-20.969	0
β_3	-38.206	2.5935	-14.731	0
β_2	-4.1879	0.39181	-10.688	0
β_1	2415.6	2.5927	931.7	0

Table F.3: Batch 2's partial t test results.

Coefficient	Estimate	Standard Error	t-statistic	p-value
β_7	-135.6	6.5267	-20.777	0
β_6	0.063305	0.7951	0.07962	0.94
β_5	29.846	6.5812	4.535	0
β_4	-1.4642	0.7977	-1.8355	0.07
β_3	44.994	6.5032	6.9188	0
β_2	5.7153	0.79516	7.1877	0
β_1	3024.1	6.2887	480.88	0

dependent on the cost of making a type 1 error, or of rejecting the null hypothesis if it were true[10]. Each batch's p-value for β_7 was far lower than any reasonable level of significance, which could be the traditional 0.05. This would infer that the interactions between the treatment group and displacement of a sample in four point bending was statistically significant. The null hypothesis is rejected due to there being strong evidence that this interaction exists and is negative.

Table F.4: Batch 3's partial t test results.

Coefficient	Estimate	Standard Error	t-statistic	p-value
β_7	-147.36	5.3335	-27.628	0
β_6	4.1213	0.74845	5.5065	0
β_5	9.4241	5.3219	1.7708	0.08
β_4	3.806	0.76098	5.0014	0
β_3	32.12	5.3204	6.037	0
β_2	-1.6628	0.7606	-2.1862	0.03
β_1	2746.2	5.3852	509.95	0

F.2 N-way ANOVA

The non-regression ANOVA model output is shown below, indicating that the pairs and treatment were both significant factors.

Analysis of Variance					
Source	Sum Sq.	d.f.	Mean Sq.	F	Prob>F
UpDown	28260628.1	1	28260628.1	3.66	0.0846
Fluences	17508680412.1	10	1750868041.2	227	0
Pair	81287231.6	1	81287231.6	10.54	0.0088
UpDown*Fluences	330711140.8	10	33071114.1	4.29	0.0154
UpDown*Pair	157550.1	1	157550.1	0.02	0.8892
Fluences*Pair	266654799	10	26665479.9	3.46	0.0316
Error	77129201.8	10	7712920.2		
Total	18292880963.4	43			

Figure F.1: ANOVA table for the bending moduli data.