

PROJECT NUMBER: PMR 1501



Design of a System for in-situ Measurements of Semiconductor-Catalyst Under Strain

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Abstract

Rising concerns over global warming and fossil fuels creates a need for alternative energy sources. Hydrogen, the most abundant chemical element in the universe, can be harnessed through solar-driven water electrolysis by a photocatalyst material. The project team designed, manufactured, and tested a device in which a photocatalyst material can be mechanically strained to improve its energy conversion efficiency. The device consists of an electrically conductive substrate, a straining mechanism, and a reaction chamber. The electrically conductive substrate, made of a metal-filled polymer, acts as an electrode for the photocatalyst. The straining mechanism was designed to bend this substrate and transfer strain to the photocatalyst over a range of -2 to 2%. The reaction chamber was created to house the substrate and mechanism while allowing light to reach the photocatalyst. All components were designed to be chemically resistant to one molar sulfuric acid, in which the reaction will be conducted.

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1.0 Introduction

1.1 Project Description

The purpose of this project is to design and manufacture a device that will allow students to conduct Hydrogen Evolution Reactions (HER). A Hydrogen Evolution Reaction produces hydrogen through water electrolysis, which is the process of separating water into hydrogen and oxygen. The overall chemical equation that describes water electrolysis is shown below.



For this reaction to occur, there must be a catalyst, electrode, electrolyte, and a source of energy. Current methods utilize platinum for the catalyst and electrode, salts or acids for the electrolyte, and either fossil fuels or electricity for the source of energy. Due to the rarity of platinum, making it one of the most expensive metals (\$974.16/oz)¹, the process is very expensive. After conducting research, it has been determined that Molybdenum Disulfide (MoS₂) can serve as a less expensive substitute for the catalyst. MoS₂ is a semiconductor that exhibits photovoltaic behavior. Since the catalytic properties of MoS₂ can be amplified under strain, the function of this device is to apply strains of up to 2% (tension and compression) to the MoS₂.

A substrate for the MoS₂ is required and will be made of a combination of a polymer and metal that is electrically conductive and chemically resistant. This substrate will be in the form of a rectangular beam that also has a similar Young's Modulus to the MoS₂ to allow strain to be transferred. The beam must be able to withstand the applied strains without any plastic deformation, so that it can be used for multiple tests.

It is possible to directly perform hydrogen electrolysis at the surface of MoS₂ by exposing it to light. In this case, the photo-excited charges in the MoS₂ have enough energy to cause the desired photo electrochemical reaction. A diagram of the envisioned process is shown below in Figure 1.

¹ Price charts. (n.d.). Retrieved April 18, 2016, from <http://www.platinum.matthey.com/prices/price-charts>

² Brenda Johnston, Michael C. Mayo, Anshuman Khare, Hydrogen: the energy source for the 21st century, Technovation, Volume 25, Issue 6,

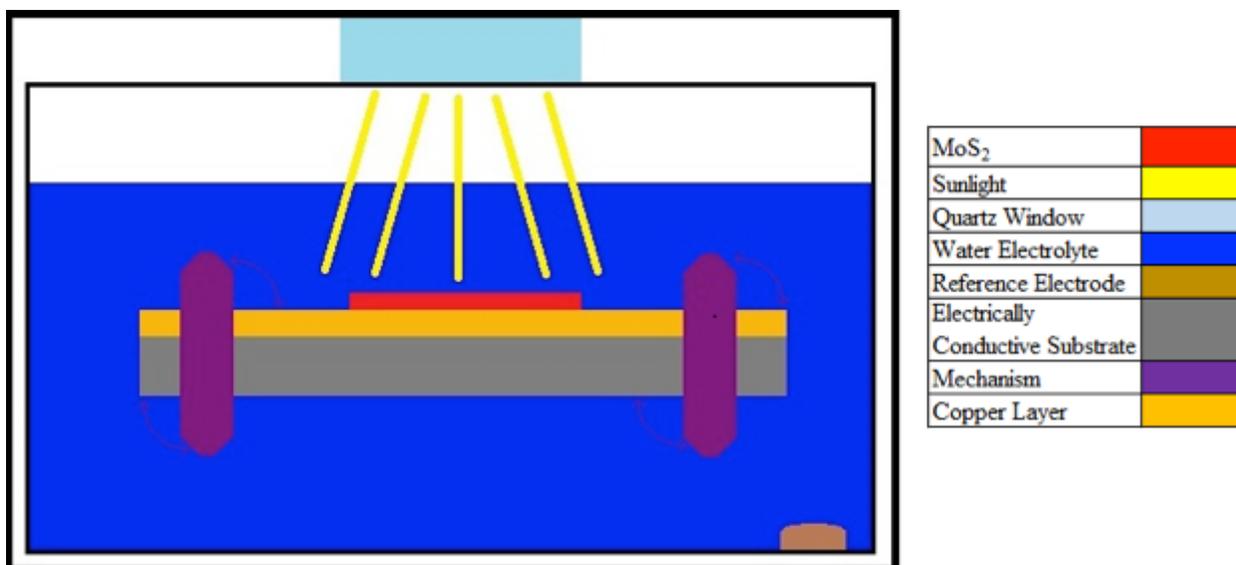


Figure 1: Photo Electrochemical Reaction

MoS₂ is sensitive to applied strain, allowing for strain engineering of its catalytic properties. Applied strain could increase the efficiency at which hydrogen is produced. There are scientific reports that theoretically define the properties of MoS₂ under strain; however, there is a lack of experimental validation. The goal of this project was to create a device to strain the MoS₂ at varying levels so the catalytic properties could be evaluated to determine the most effective condition for electrolysis. The strain on the substrate will be transferred to the catalyst, which is stationed in the center of the substrate. This transferred strain is different from the strain induced by the mechanism and must be measured using a Raman Spectrometer.

1.2 Motivation

Hydrogen is one of the cleanest energy sources available, for it produces almost no exhaust and is the only product of the reaction is water². Currently, 95% of hydrogen is produced using wood, natural gas, and oil, leaving a large carbon footprint³. There is growing interest in minimizing that footprint and creating a sustainable method for hydrogen production. An envisioned hydrogen cycle under ideal conditions is shown in Figure 2.

² Brenda Johnston, Michael C. Mayo, Anshuman Khare, Hydrogen: the energy source for the 21st century, Technovation, Volume 25, Issue 6, June 2005, Pages 569-585, ISSN 0166-4972

³ Ogden, J.M. (1999). "Prospects for building a hydrogen energy infrastructure". Annual Review of Energy and the Environment 24: 227-279. doi:10.1146/annurev.energy.24.1.227

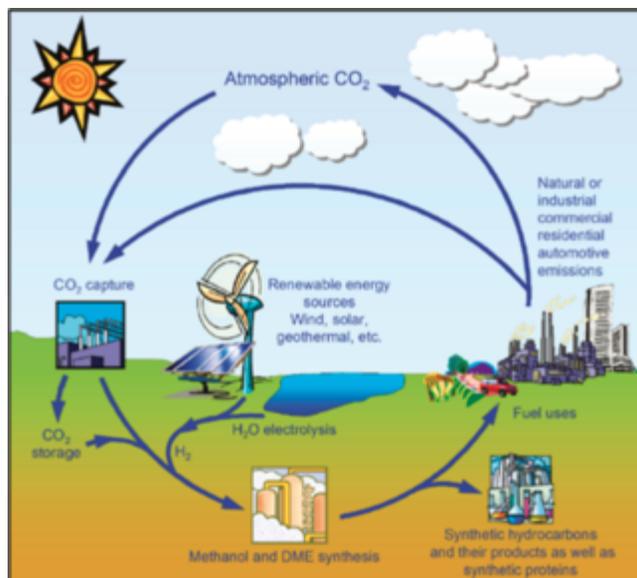


Figure 2: Hydrogen Cycle

At this time, the most common method for hydrogen production is steam reforming. This method separates superheated vapor and methane creating hydrogen gas and carbon monoxide. Hydrogen Evolution Reactions can also occur using electricity as the energy source. This method sends an electric current through the electrodes into the water, separating it into hydrogen and oxygen. The hydrogen will appear at the cathode while the oxygen will generate at the anode. This method is shown below in Figure 3.⁴

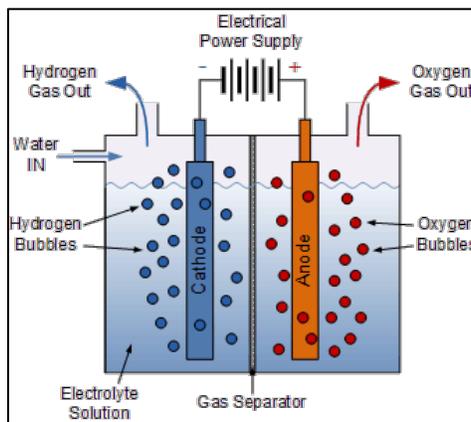


Figure 3: Water Electrolysis

Water electrolysis also utilizes catalysts to enhance the rate at which the reaction occurs. To determine what materials could serve as a good catalyst, Sabatier Principle is used. Sabatier

⁴ Hydrogen Energy is Free Energy from the Sun (n.d.) Retrieved March 28 2016, from <http://www.alternative-energy-tutorials.com/energy-articles/hydrogen-energy.html>

Principle states that the hydrogen binding energy of the material should be neither too strong nor too weak. If it is too strong, the reacting species will quickly cover the whole surface of the catalyst causing the reaction to cease. If the binding energy is too weak, the rate at which the reaction occurs is greatly decreased. The binding energy can be plotted versus the current density to find the ideal catalyst, which is directly in the center of the x-axis and high on the y-axis. This graph is called a volcano plot due to the shape of the curve. Figure 4 displays this relationship.⁵

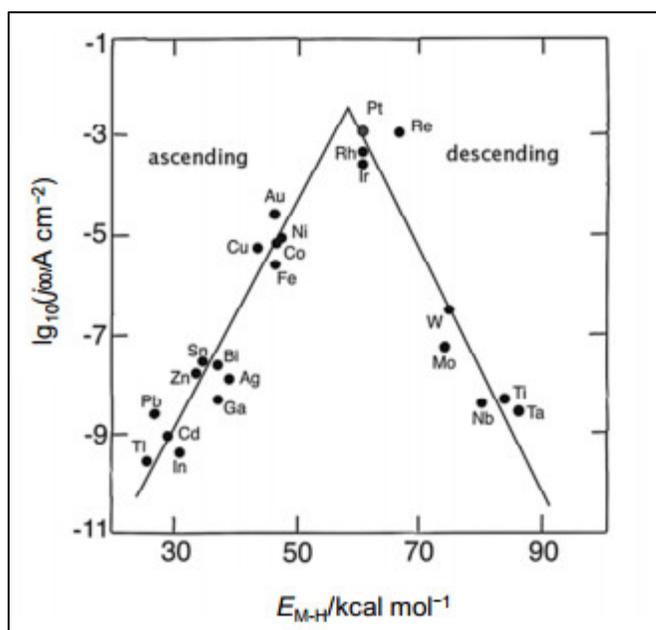


Figure 4: Volcano Curve

It is clear that platinum (Pt) is the best catalyst, which explains why it is the most predominant catalyst used. Platinum can also serve as the electrode in water electrolysis. Electro-catalysts can function either on the surface of the electrode or be the electrode. Water electrolysis is much cleaner than steam reforming; however it is around four times more expensive⁶. Platinum alone is one of the rarest metals on the planet making it very expensive. This is the major obstacle in the path of commercialization of this process. Not shown on this chart is MoS₂. Mo is shown but is not near the optimal position on the chart. The combination with S₂ increases its effectiveness as a catalyst, but it is the applied strain that makes it an optimal choice. Applying strain to the MoS₂ can tune its binding energy positioning it around the same area as platinum on the chart.

⁵ Quaino, P. (2014, June 13). Volcano Plots in hydrogen electrocatalysis – uses and abuses. Retrieved March 28 2016, from <http://www.beilstein-journals.org/bjnano/single/articleFullText.htm?publicId=2190-4286-5-96>

⁶ Burtain, A. (2015, July 7). Hydrogen Production. Retrieved November 10, 2015, from <http://www.planete-energies.com/en/medias/explanations/hydrogen-production>

2.0 Background

2.1 Mechanisms Used to Apply Strain

Experiments have previously been conducted to test strain on materials, though the specific goals of these experiments differ from that of this project, aspects of each can be taken to inform the design for the straining mechanism. Many of these experiments have created devices to stress and strain one specific material. For example an experiment run by engineers from Ohio State University, the Material Science Division of Alcoa Technical Center, and Seoul University tested the large strain of sheet material⁷. The purpose of this experiment was to test and better understand the different types of buckling on sheet metal. A new method of applying tension and compression was created during this experiment. This approach uses solid flat plates as constraints to pinpoint buckling to the location desired. The solid plate design was chosen over a fork design since it is easier to machine and more durable. A drawing of the flat plate idea is shown in Figure 5 while Figure 6 depicts the final machine that was used to stress and strain the sheet metal.

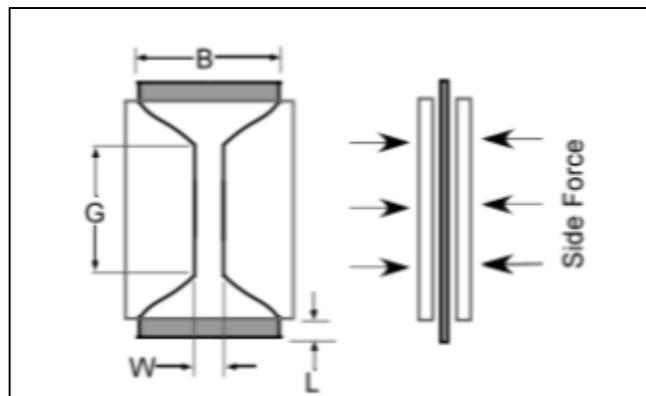


Figure 5: Solid Plate Strain/ Compression Design

⁷ V Körstgens, H.-C Flemming, J Wingender, W Borchard, Uniaxial compression measurement device for investigation of the mechanical stability of biofilms, Journal of Microbiological Methods, Volume 46, Issue 1, 30 July 2001.

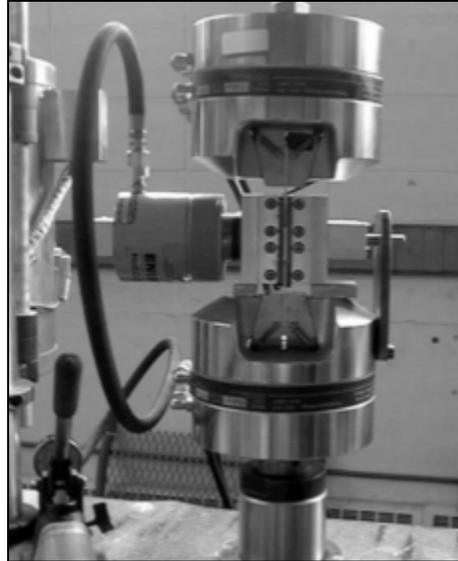


Figure 6: Flat Plate Stress Compression Device

The overarching goal this project was to reach the required strains and to have a uniform distribution of strain and stress throughout the specimen. Even though this is a much larger scale of uniaxial stress and strain, this experiment gives insight into development techniques and different types of bending. This experiment also represents a proven example of using uniaxial forces to generate strain and stress. Knowing that a device using this type of uniaxial force already exists helps us know that this would be plausible for a final design.

Another experiment performed on a smaller level was conducted by engineers at Washington University in St Louis working with a three dimensional manipulation of carbon nanotubes under a Scanning Electron Microscope⁸. To manipulate the material under the microscope, a device was needed that had to be small and very precise. This device had a stage that could move up to 6mm in the X, Y and Z direction and rotate 360 degrees, so that the nanotubes could be fully examined. The device that was used is shown below in Figure 7. Piezo tubes were used for supports that are 12mm long with a 6mm diameter. The whole device occupies a space of 50cm³.

⁸ Yu, M., Dyer, M., Skidmore, G., Rohrs, H., Lu, X., Ausman, K., Ruoff, R. (1999). Three-dimensional manipulation of carbon nanotubes under a scanning electron microscope. *Nanotechnology*, 244-252.

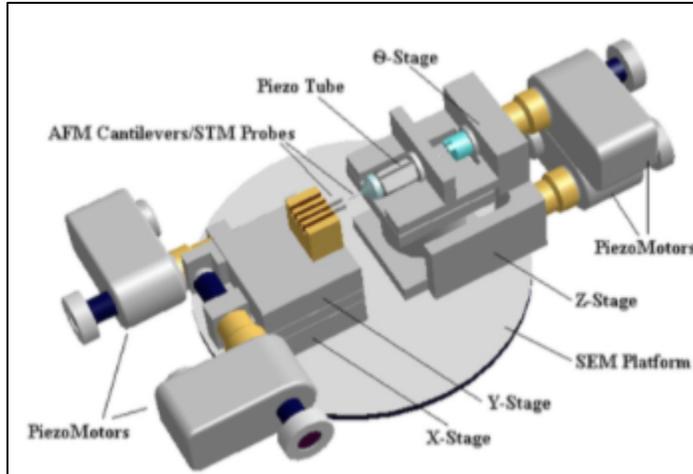


Figure 7: Three Dimensional Manipulation Device

This device was created to change the object orientation and move it in three linear directions, which is much more complicated than the device that will be created in this project. This device was created in a smaller scale and helps us realize the possibility of success of a bending device.

Another example of the effect of torsion and bending is an experiment conducted on Casimir attraction coupled with the pull-in instability of an electrostatic nano-actuator.⁹ An electrostatic torsional actuator was used in this experiment shown in Figure 8. This device was created using two elastic torsional nano-beams over two fixed substrate electrodes. The beams are supported on each end and when there is a voltage applied to the plate, the plate would rotate towards the electrode. This rotation was used to bend the material. This is another design idea that could be taken into consideration when making the device for this project.

⁹ Tadi Beni, Y., M. Abadyan, and A. Koochi. "Effect on the Casimir Attraction on the torsion/bending coupled Instability of Electrostatic Nano-Actuators." *Physica Scripta* 84 (2011)

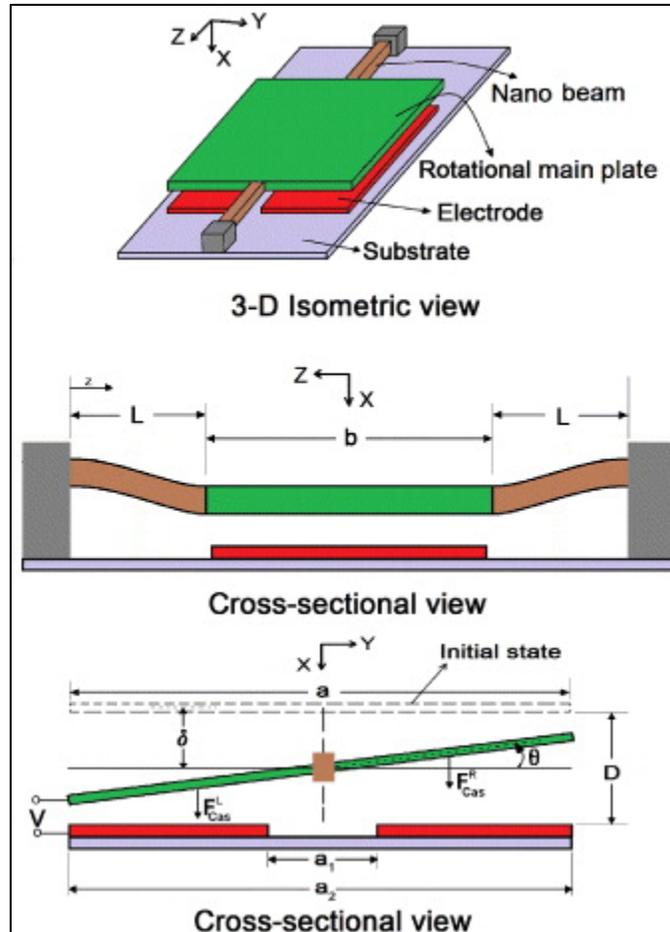


Figure 8: Electrostatic Torsional Actuation Bending Device

Lastly a device was created using pure bending in 2015 to test the mechanical limits of manufacturing loads for flexible electronics¹⁰. This device was created to be an autonomous and miniature pure bending device to determine the results on a microscopic level. This paper goes through the process of determining which method to use for bending, which is very similar to what will need to be done for this project. The paper goes through two, three, and four point bending methods and how they will affect the specimen. These methods were discarded during this experiment since the entirety of the specimen will be an electronic device. The point-bending methods put extreme pressure on singular points that can cause the specimen to deform. Due to these defects, pure bending was chosen for this device. In the beginning pure bending was tested using sliders, which resulted in friction that caused the bending to not be pure. Frictionless air bearings were created to aid this process and to ensure the process as pure as possible.

The design, shown in Figure 9, shows that clamps were used to attach the bar and cause the bending. Both of the clamps have linear actuators that translate the force to the specimen.

¹⁰ Hoefnagels, J. P. M., A. P. Ruybalid, and C. A. Buizer. "A Small-Scale, Contactless, Pure Bending Device for in-Situ Testing." *Experimental Mechanics* 55.8 (2015): 1511-24. Web.

Interestingly one of the clamps has an additional actuator to compensate for the turning force. This example of a bending design and the process to create the bending design are very useful to understand where to begin for this project.

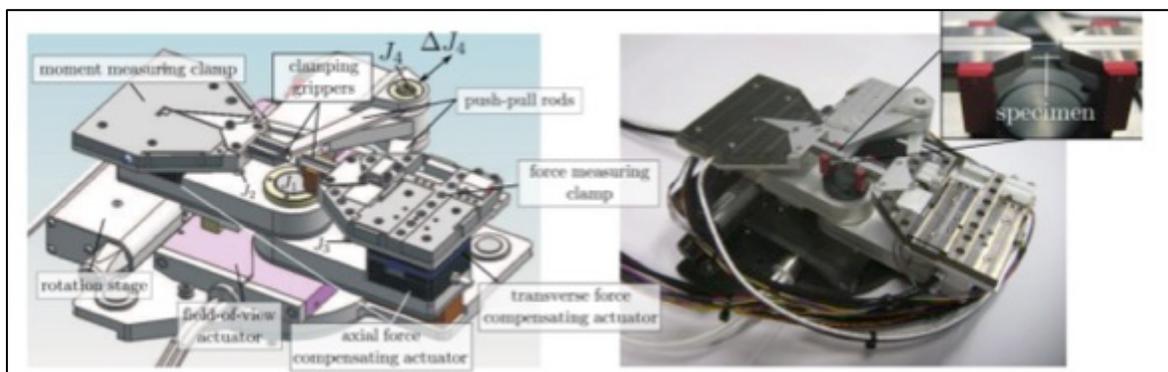


Figure 9: CAD Model and Actual model of electronic bending device

2.2 Molybdenum Disulfide

Experiments have been conducted on MoS₂ to determine the effect of strain on its catalytic properties. A report from Yonsei University¹¹ compares the electrochemical activities of unstrained MoS₂ and MoS₂ strained at 0.005%, 0.01% and 0.02%. These percentages are the strain applied to the substrate, not the actual strain of the MoS₂. The researchers hypothesize that tensile strain would increase the electro-catalytic performance of MoS₂ Nano-sheets. It was discovered that the strain induced caused a steeper polarization curves than without strain. The applied strain to the MoS₂ also had increased electrochemical activities towards the hydrogen evolution reaction, as well as creating a very efficient electro-catalysis system for evolving hydrogen. The justification for this claim is provided through conclusions made from dynamic band theory. Figure 10 depicts the overall concept of the hypothesis.

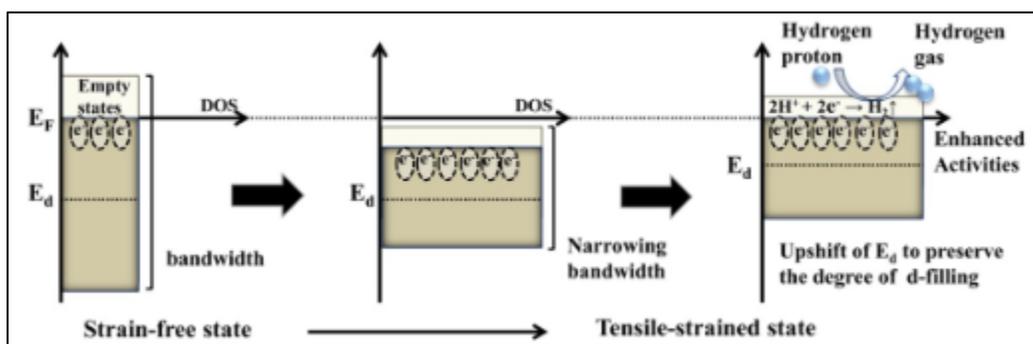


Figure 10: Increase of Electrochemical Activity with Strain

¹¹ Baik, H. (2014). Efficient Hydrogen Evolution by Mechanically Strained MoS₂ Nanosheets. *Langmuir*, 30(32), 9866-9873. doi:10.1021/la501349k # Tadi Beni, Y., M. Abadyan, and A. Koochi. "Effect on the Casimir Attraction on the torsion/bending coupled Instability of Electrostatic Nano-Actuators." *Physica Scripta* 84 (2011)

This increase in catalytic activity is what will be replicated in this project. The goal is to create a device that will apply both tensile and compressive strain, so that the relationship between strain and catalytic properties can be fully investigated. For this experiment, the MoS₂ specimen must be in monolayer or bilayer form due to increasing stiffness as layers increase. The more rigid the material is, the more difficult it will be to strain. This could cause the applied strain to become non-uniform and only transfer to the lower layers, leaving the upper layers more susceptible to slippage.

A different study conducted at Vanderbilt University examined the effect of strain for the optical properties of MoS₂¹². Strain was induced through four-point bending while the optical properties were evaluated through Raman Spectroscopy. The process is shown in Figure 11.

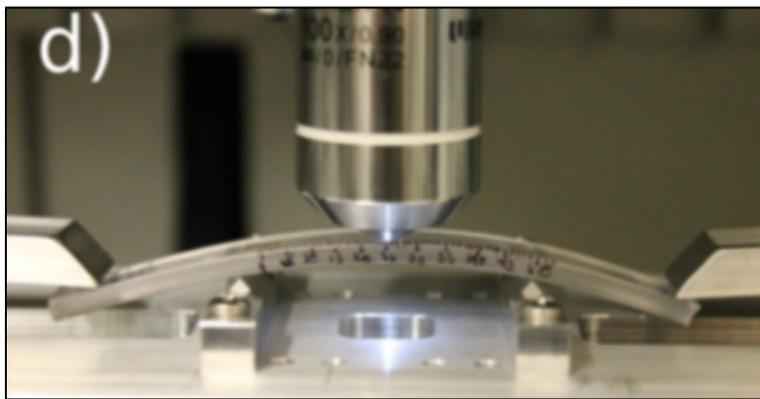


Figure 11: Four Point Bending Under the Raman Spectrometer

This report shows the influence of 0-2.2% uniaxial mechanical strain on the phonon spectra and band structures of monolayer and bilayer MoS₂. In this experiment ideal fixturing between their polycarbonate beam substrate and MoS₂ specimen is assumed. This would be the ideal case but is not applicable to real world experimentation. The slippage is determined by how similar the Young's Modulus are for both the MoS₂ and the substrate. The closer the numbers are the less chance there is for slippage to occur.

In the study from Yonsei University¹¹, small amounts of strain were applied to MoS₂ in order to observe a change in catalytic performance. Unfortunately for the purposes of this project, the researchers did not evaluate strain transfer with Raman spectroscopy. Even though, they reported an overall improvement of catalytic properties, their work is not sufficient support for strain engineering of MoS₂. In the study from Vanderbilt University¹², 2.2% strain was applied to the MoS₂. This strain was confirmed with in-situ Raman measurements, however, the specimen was in dry conditions outside of catalytic activity. This study also fails to describe how strain transfer is affected by submersion in 1 molar sulfuric acid.

¹² Bolotin, K. (2013). Bandgap Engineering of Strained Monolayer and Bilayer MoS₂. *NANO Letters*, 13(8), 3626-3630. doi:10.1021/nl4014748

3.0 Project Specifications

3.1 Components

All three components of the straining device each have their own requirements in order for the experiment to properly occur. Below are descriptions of the components and detailed descriptions of what is required for each part.

Beam

The substrate will be created in the form of a rectangular beam. A rectangular beam is very simple and can be replicated with ease. The main purpose of this beam will be to serve as the electrode while transmitting strain to the MoS₂. To serve as the electrode the beam must be electrically conductive. Testing the beam with a multimeter will be the first test conducted on experimental beams for it is the most important quality of the beam. The dimensions and properties of the beam will affect those of the mechanism and chamber since the chamber will house the beam while the mechanism has to be specifically made work with the substrate used. The beam is required to have enough area so the MoS₂ can be applied easily, and also be thin enough to transfer strain to the MoS₂. Though the beam must also be thick enough so that there will be no buckling or plastic deformation. Plastic deformation of the beam would negatively impact the results of tests making it unusable. There must be space to accommodate a strain gauge so that the stresses on the beam can be recorded. Additionally the size of the beam will need to fit the mechanism that is chosen: longer if the mechanism is outside the chamber and shorter if the mechanism is inside the chamber.

The beam is required to be submerged in sulfuric acid. To enable this to happen the materials used for the beam must be chemically resistant.

Mechanism

The purpose of the mechanism is to apply compressive and tensile strain to the beam. Many ideas were initially considered for how the mechanism could operate. First and foremost the mechanism must be able to induce the necessary stress and strain. It also must be able to fit in the Raman spectrometer. The Raman spectrometer, shown in Figure 12, only has about one and a half inches of clearance for the device to sit. This leaves two options for the mechanism, either makes it small enough so it can fit underneath the spectrometer or position it outside of the chamber where there is less of a space limit.

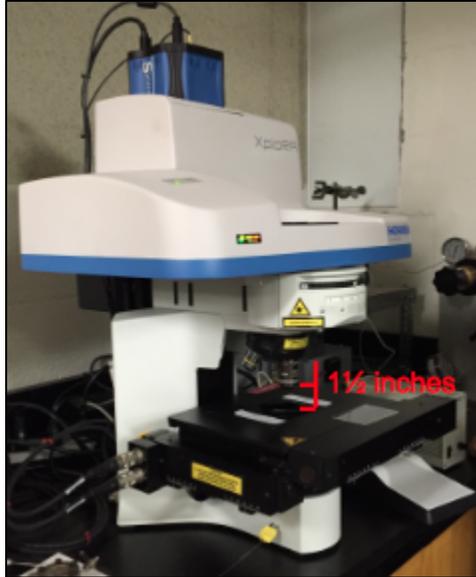


Figure 12: Raman Spectrometer at WPI

The mechanism must be chemically resistant for a large portion of the mechanism will be inside of the chamber and submerged in the acid. There is still a possibility that the parts outside of the chamber could come into contact with the acid so in the interest of longevity and safety the entire mechanism will be made of chemically resistant material.

Longevity and high reliability are two of the most important traits for the mechanism. One of the main goals for this project is to make sure all parts of the device will work at peak performance for as long as possible. The mechanism should apply forces in the most simplistic manner and be as straightforward as possible. The fewer moving parts and variables that are involved, the longer the mechanism will last and the easier it will be to replicate once it does finally breakdown. The cross section and length of the beam will also influence the mechanism for it will determine the shape and size of the grips as well as how strong the mechanism needs to be. There is a small amount of strain applied, but it will require the mechanism to be highly precise. Two percent strain equates to very little motion of the mechanism. For example, if the length of the bar is 4 inches, four multiplied by two percent is .08 inches. This would be excluding strain efficiency, which is a factor relating the actual and the transmitted strain.

The strain applied to the beam through the mechanism must be adjustable from outside of the chamber, which will ensure superb accuracy. It would be very impractical for the controls to be inside the chamber since the operator would have to reach inside of the chamber which would create many variables that could influence the testing and also, given the small working dimensions due to Raman Spectrometer, it would be almost impossible.

Chamber

The final component of this device will be the chamber that will contain the electrolysis reaction. This chamber will house the beam and parts of the mechanism as well as the acid needed for the reaction. If parts of the mechanism or beam are outside of the chamber, the

chamber will also need to be shaped to accommodate these, while keeping the reaction water and airtight. Dynamic seals must be created for parts of the mechanism or the beam that would need to move outside of the chamber so they could move while keeping the chamber liquid and airtight. If the chamber is not liquid and airtight there could be a leakage of acid, and other gases could enter the chamber influencing the reaction taking place. Figure 13 shows an example of a chamber that is currently used in experimentation.



Figure 13: Example of an Existing Chamber

For this experiment, there will be a separate lid that can be removed and resealed for each experiment. The new chamber will need to have the five ports with the same dimensions that the current device has so that there will not have to be any redesigns for parts outside of this device. Having parallel dimensions is important so the measuring devices that go into the ports do not have to be changed to keep the experiments running. These ports will also have to be water and airtight.

The entire chamber needs to be chemically resistant so the reactions on the inside do not affect the material. The material must be able to be resistive to the sulfuric acid and similarly to the mechanism, there is a wide range of materials to choose from. The material must also be able to be easily machined while also being very durable. The material chosen should be able to withstand years of experimentation before the chamber fails and needs to be replaced.

Lastly the chamber requires a window with an approximate diameter of 1.5 cm to allow the required light in for the catalytic reactions. This window would be positioned in the center of the lid to the chamber. The material for the window should have to be completely clear, chemically resistant, as well as air and water tight.

3.2 Requirements

To clarify what would be an excellent design from a satisfactory design, a table has been created to describe what should be accomplished for each part of the device. These are the standards that the device must meet, aiming to get everything in the excellent category.

	Excellent	Good	Satisfactory
Beam	<ul style="list-style-type: none"> • $\rho (\Omega) \leq 1.0 * 10^{-6}$ • At least 2% elastic deformation • 2 Machining Operations • Sustain up to 12hrs in acid • $E > 650$ MPa 	<ul style="list-style-type: none"> • $\rho (\Omega) \leq 1.0 * 10^{-3}$ • Has up to 2% of elastic deformation • 3-4 Machining Operations • Sustain up to 10hrs in acid • $1 \text{ MPa} < E < 650$ MPa 	<ul style="list-style-type: none"> • $\rho (\Omega) \leq 10$ • Does not get to 2% of elastic deformation • 4+ Machining Operations • Sustain less than 6hrs in acid • $E < 1$ MPa
Mechanism	<ul style="list-style-type: none"> • Bidirectional • 2 moving parts • It is chemically resistant (only applies for in chamber mechanism) • 0.1% digits precise • Easy to operate 	<ul style="list-style-type: none"> • Unidirectional • 3-5 moving parts • Partially chemically resistant • .2% digit precise 	<ul style="list-style-type: none"> • Unidirectional • 5+ moving parts • Not chemically resistant • 0.5% precise • Hard to operate
Chamber	<ul style="list-style-type: none"> • Chemically resistant for 12+ experiments • Flat bottom • Lid comes out • Space for 5 ports • Air/water tight • Fits between stage and lense 	<ul style="list-style-type: none"> • Chemically resistant for 6-12 experiments • Space for 3 ports • Water tight • Stage has to be taken off from Raman Spectrometer 	<ul style="list-style-type: none"> • Chemically resistant for less than 6 experiments • Round bottom • Space for less than 3 ports • Not sealed • Does not fit under Raman

4.0 Methods

Figure 14 is a flowchart depicting the process used in creating the full device. It began with the initial background research then is split into two separate design categories, the beam and the mechanism. Once the dimensions of the beam were determined, the dimensioning the mechanism could begin. In the meantime design specifications for the mechanism were determined, ideas formulated and multiple designs emerged. These designs were narrowed to two before using a decision matrix to find the best design. Once the dimensions were finalized, the chamber dimensions could then be calculated to house the entire reaction while still fitting under the Raman Spectrometer. After both the beam and the mechanism were completed and manufactured, separate development processes and test that did not influence the other component.

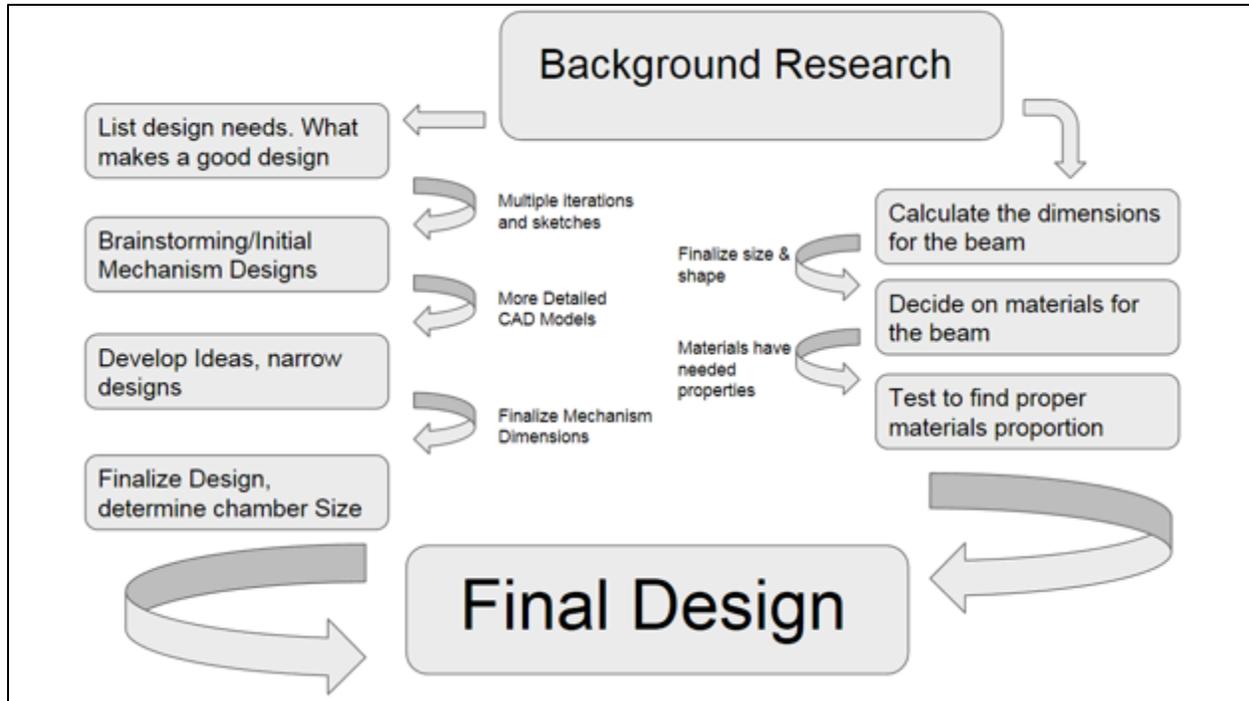


Figure 14: Process Design Chart

5.0 Initial Designs and Mechanical Analysis

To begin the design process, time was spent brainstorming ideas and drawing rough sketches of functional devices. All of the initial mechanism ideas incorporated a different method of applying the strain to the MoS₂, leading to multiple unique complete design ideas. Once the initial brainstorm was finished, ideas began to be refined. These designs are much more developed and have more detail than the original iterations.

5.1 Beam

Figure 15 shows the Loading force P and critical buckling force P_{Critical} varying with the cross section in axial loading. In this case the beam is most easily designed with a square cross section since buckling occurs in the direction of lower moment of inertia ($\min [I_x, I_y]$). The red curve represents the absolute difference between loading force and buckling force. The blue, green, and red curves show the value of the square side length (c), needs to be greater than the point at which the red curve equals zero. This comes from the loading force depending on the cross sectional area and the critical buckling force depending on the moment of inertia of the cross section.

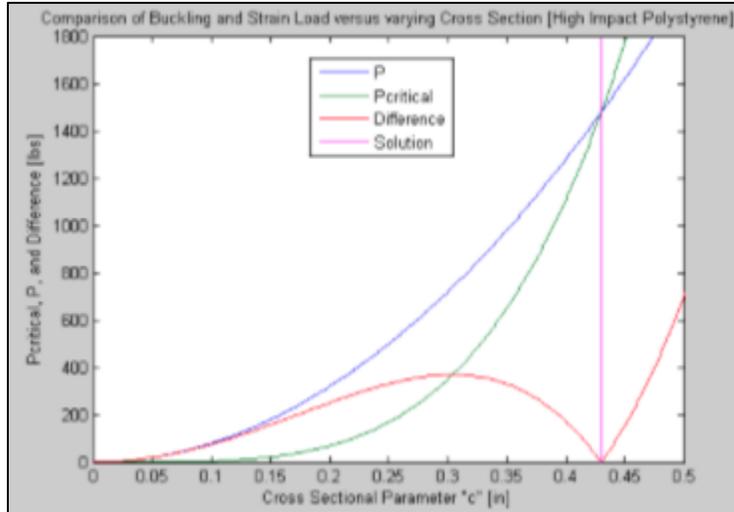


Figure 15: Buckling and Displacement Force vs. Cross Section Properties

In a recent study¹³, it was discovered that a polymer with a Young’s Modulus of less than 1 MPa could transit about 0.5% of the total strain to a sample of MoS₂, while a polymer with a Young’s Modulus of 650 MPa could transfer about 60% of the total applied strain. Figure 16 is a very rough logarithmic model to describe the behavior of strain transfer efficiency. From the previous study⁶, it is clear that the closer the Young’s Modulus of the polymer metal combination is to the Young’s Modulus of the MoS₂, the more strain will be transferred. A Raman Spectrometer will monitor the amount of strain transferred.

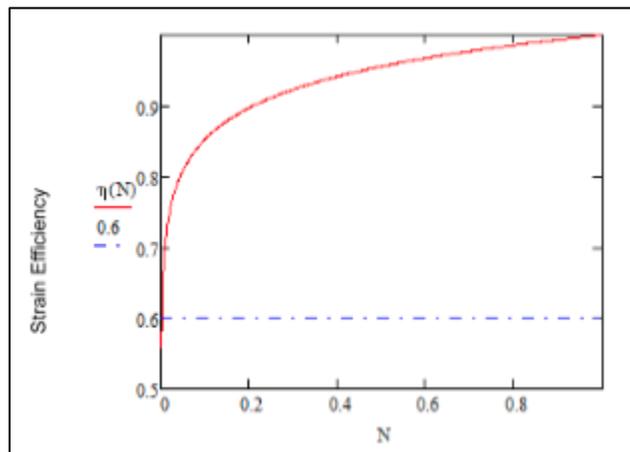


Figure 16: Strain Efficiency vs. (Es/Em) = N

5.2 Mechanism

To begin this project there were multiple different methods in consideration for applying strain to the beam. These methods included, a balloon system, hydraulic actuator, uniaxial

¹³ Liu, Z., Amani, M., Najmaei, S., Xu, Q., Zou, X., Zhou, W., . . . Lou, J. (2014). Strain and structure heterogeneity in MoS₂ atomic layers grown by chemical vapor deposition. *Nature Communications Nat Comms*, 5246-5246.

loading and four-point bending. Though all the ideas on this list could be applied, ultimately four-point bending, shown in Figure 17, and axial loading, shown in Figure 18, were chosen to create detailed designs for due to their simplicity and effectiveness. These methods have both been extensively used in experiments as a means of applying strain. For axial loading, the mechanism would apply tension and compression to the cross-sectional area of the beam. This can be achieved by applying the forces on one or both sides. If the force is only applied to one side of the beam, there are only a few components that need to move to put strain on the beam. In addition this design would have one side fixed to the chamber, limiting the parts that would need to leave the chamber and would need to have air and watertight seals. If the force is applied on both sides, the beam will be able to be uniform and easy to recreate. The beam set up for the four-point-bending would be almost identical to the two-sided axial loading. The entire mechanism can be outside of the chamber and could be bidirectional. Both uniaxial loading and four point bending designs could work, but to create and maintain the device, one with the least amount of moving parts and variables would be best. These designs will be discussed in further detail in the Design section below.



Figure 17: Uniaxial Loading

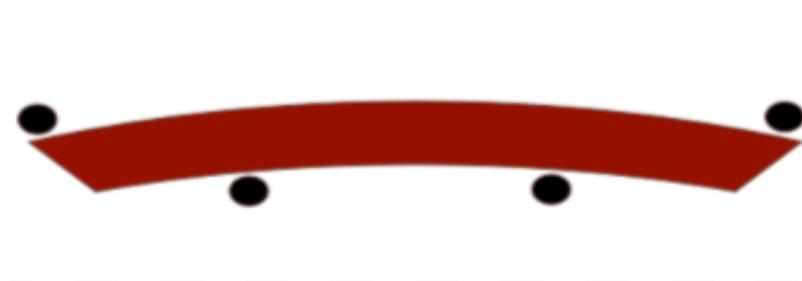


Figure 18: Four-Point Bending

5.3 Chamber Design

Initial chamber designs were limited because the chamber needed to house the mechanism, so getting a rough idea of the size of the mechanism would give an idea of the size the chamber would need to be. Something that was considered for the beginning of the chamber design was the material that would be necessary for the chamber. These material requirements, listed in the requirements section, explain the extensive requirements the chamber must fill. A material that fits these criteria is Teflon, for it is chemically resistance and could be machined into the shape required. Another important factor of the chamber is that it will be able to be keep

its chemical resistance while being submerged in water or acid for long period of time. The chamber will be designed with the expectation that it will be used for many years, which means it will need to be strong enough to go through repeated use and washes of chemicals.

Also a material for the clear window was researched during this stage. Two different materials were considered for this window, glass and quartz. Both of these materials are clear and will let the needed amount of light through, are chemically resistant, and are easy to acquire.

6.0 Initial Mechanism Designs

The mechanism is responsible for applying the force on the beam. There is room for flexibility for how this force can be applied. After conducting research, multiple methods to apply force were designed and fitted to work for this experiment. This section contains descriptions and rough drawings of some the different designs for the mechanism that were originally created. It is important to note that at this point in the process dimensions had not been determined for any component of the device

Four-Point Bending

Four-point bending is a technique where there are four points of contact on the substrate, two points positioned above the beam and two underneath it. Figure 19 shows a design for a possible four-point bending mechanism.

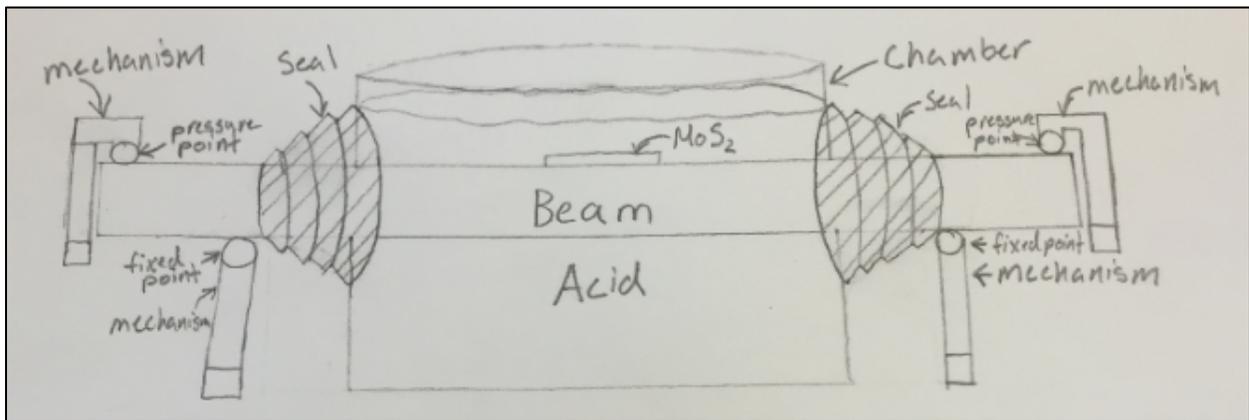


Figure 19: Four Point Bending Design

This technique is considered because it causes a uniform deformation over the beam and through the substrate. Since this is a simplistic design requiring a plain beam and simplistic mechanism. The mechanism lends itself to a simple design for the beam. The mechanism is also not constrained by the size of the chamber since the majority of it is outside of the chamber and will not corrode or be in contact with the acid.

Uniaxial Loading

Uniaxial loading is the act of applying force on an object on a single axis. This force would either be compressing the substrate in or it would be stretching it out. Having both tension and compression makes this method attractive for designs moving forward. When incorporating this method into the initial designs, this method can be applied on either one or both sides of the beam.

Two Way Uniaxial Bending

The design for two way uniaxial loading is similar to the design for four point bending. The beam is much longer than the chamber and breaks through the chamber on both sides. There are two seals in place to ensure that the chamber is still air and liquid tight even when forces are being applied to the beam. The seals must be able to adjust to the increasing and decreasing of the beam's cross sectional area when forces are applied. Clamps are then attached to each side of the beam and will be connected to a control that will move them together or apart at the same rate causing compression or tension. This design is shown in Figure 20.

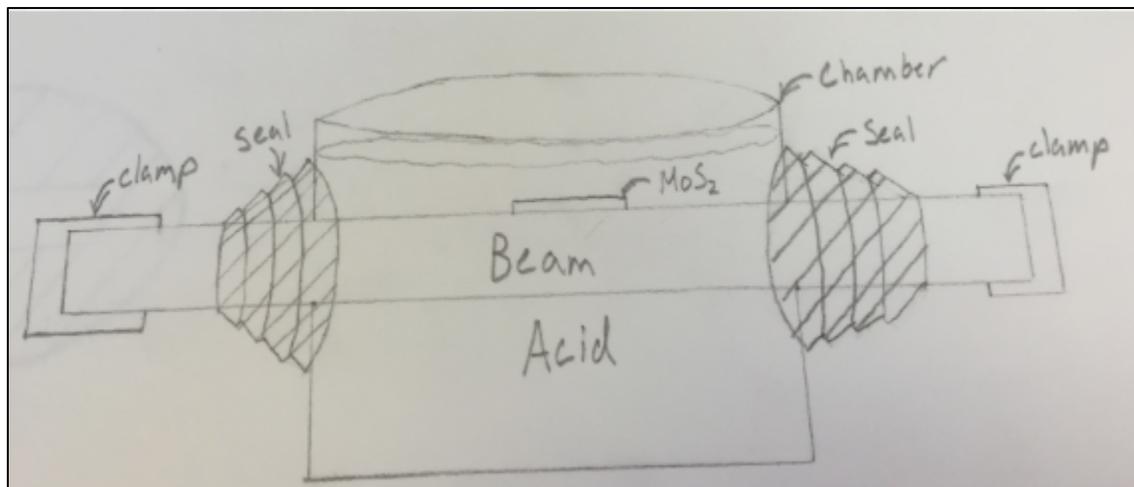


Figure 20: Outside the Chamber Four Point Bending

There are many features that make the two way axial loading an attractive design idea. First, the mechanism is entirely outside of the chamber. There would be almost no chance that any corrosion from the acid could occur on the mechanism. The beam would be very easy to design. The beam would be a rectangular prism with no holes drilled into it, for both ends would exit the chamber and be able to be clamped at the end to induce stress. This makes replicating the beam very easy for any future experiments. Lastly, with this design both tension and compression would be guaranteed to occur giving us two different types of forces causing strain on the MoS_2 .

An aspect of this design that would require much attention would be the buckling of the beam. Using the dimensions of the beam and the material properties, it would be possible to determine the amount of force that would make the beam bend. Once the force is calculated it

can be avoided since the buckling would ruin the results. Another possible issue would be the beam seals. Since the beam is rectangular, it would be likely that the seals would have to be custom made to ensure that it stays liquid and airtight.

One Way Uniaxial Bending

The other uniaxial loading device would be a one way or one-sided device. For this mechanism one side of the beam would be fixed to the wall of the chamber, while the other side would have a threaded rod worked into the beam. This threaded rod would go from the beam, leave the chamber and attached to a gear system, which would be outside the chamber. This gear system would have to then attach back to the chamber so that the gears would be able to move in and out on the threaded rod, creating tension and compression on the material. This design is drawn out in Figure 21.

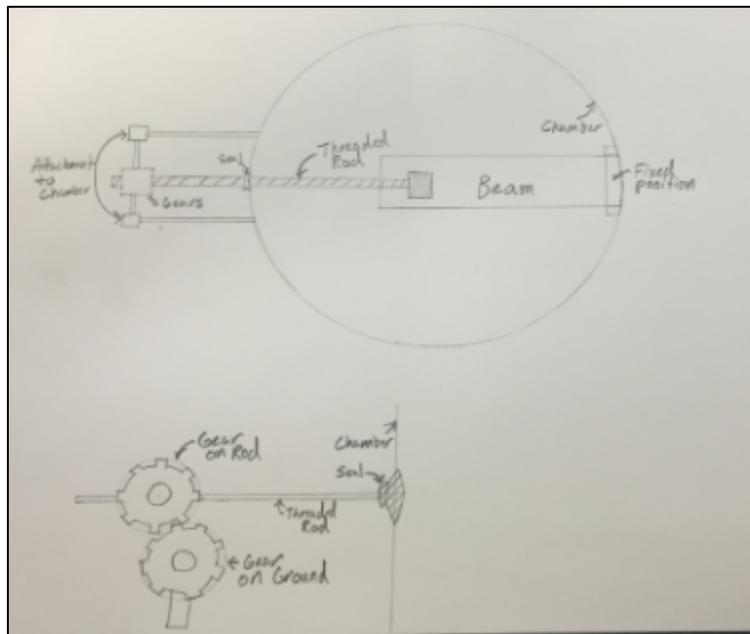


Figure 21: One-Way Uniaxial Loading

This design has many positive features. First, there would only be one spot where the mechanism would have to leave the chamber, so there would only be one location required to have a seal. Also with this design there would only be one side that would have a mechanism, so there would be fewer parts than any other design. Lastly the design would be able to put both tension and compression on the beam without having to change any parts.

Though this design has many positives, but also there are reasons to be wary of this design as well. First one would have to drill into the beam to make this design work. This would require the beam to have a pre-drilled hole for the threaded rod to enter, or it would require a mold to be created so that the beam could harden to its original shape around the threaded rod. With both of these designs for the beam it would need to be ensured that the connection between the

two would be strong enough to not break when the force is being applied to the beam in a singular spot.

Linear Pneumatic Actuator

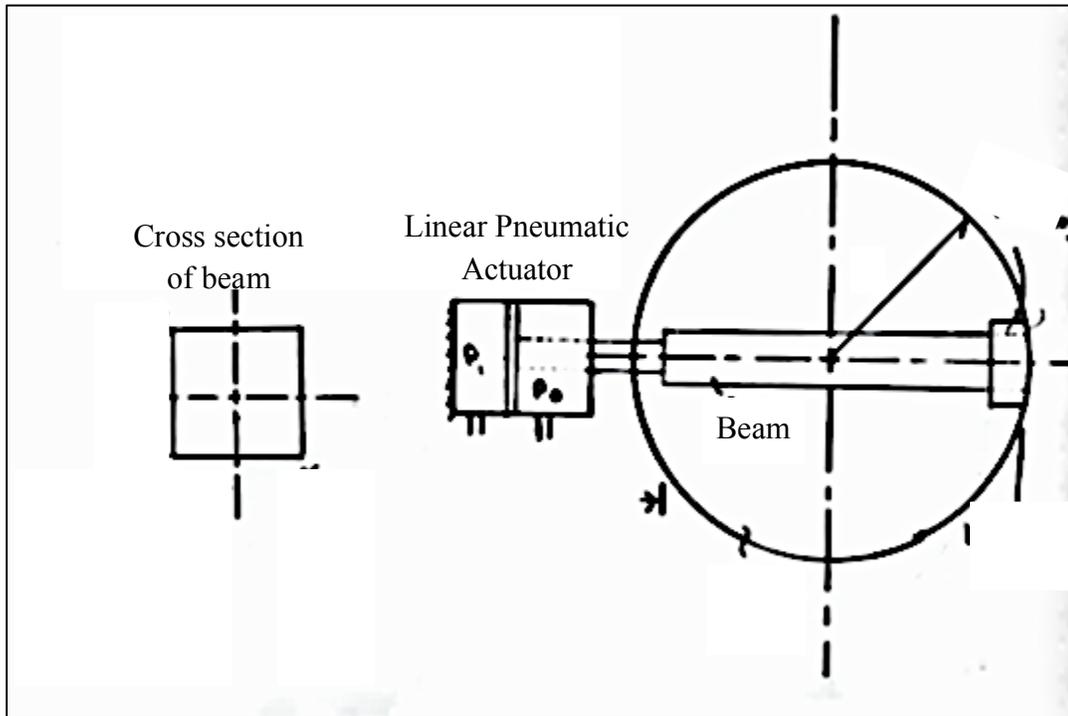


Figure 22: Linear Pneumatic Actuator

This design utilizes a hydraulic / pneumatic piston to apply either tension or compression to the beam. The beam may be grounded to the chamber to reduce the number of moving parts required to apply strain. The chamber will also need to carry the load applied to the beam if used as a ground. A frame might be required to act as ground if the load is sufficiently high. Some of the important parameters for this design are labeled in Figure 22. The Length of the beam L_b , size of the chamber r_c , pressures P_1 and P_0 , and cross sectional properties for the beam. For $P_1 > P_0$, compression will be applied to the beam. Conversely, for $P_1 < P_0$, tension will be applied to the beam.

Balloon Design

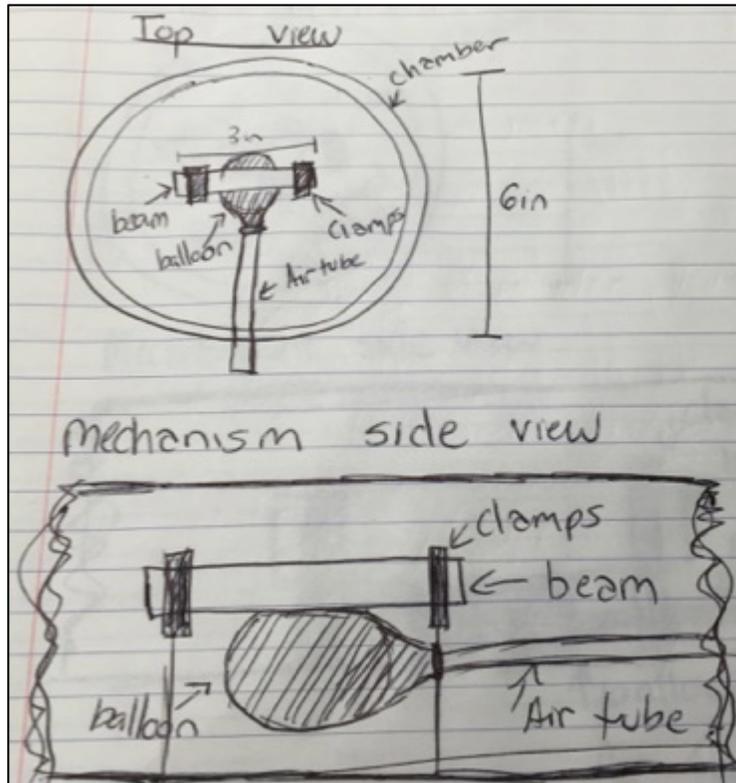


Figure 23: Balloon Design

In this design, shown in Figure 23 an air bladder would be placed under the beam and filled with air so that it will inflate and bend the beam. Since it would be air pressure that would apply the force there would only have to be an air tube leaving the chamber, and this tube would not have to move or rotate which means that the entirety of the chamber could be sealed. The bladder must be strong enough to bend the beam so that the required strain can be achieved.

Rotating Design

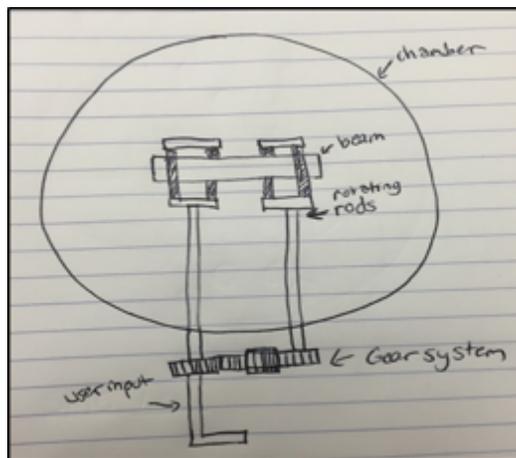


Figure 24: Rotating Design

This design contains two rods connected to a gear system that will rotate to apply pressure on the beam. Depending on which way the rods are rotated determines whether the pressure is tension or compression. The rotating rods are attached to a fixture containing two smaller rods, one above the beam and one below the beam. These rods are what make contact with the beam to cause the pressure. Figure 24 is the initial sketch of the design.

7.0 Detailed Designs and Development of Final Iterations

7.1 Beam

The substrate will be formed in the shape of a rectangular beam. This beam needs to be chemically resistant to the 1 molar sulfuric acid, elastic enough to transmit 2% strain to the MoS₂ and be electrically conductive. Due to these strict qualifications for possible materials, the group decided to pursue a path of combining materials in order to create a mixture that has combined properties. In order to ensure the beam is electrically conductive a metal will be used while a polymer will give the beam flexibility and chemical resistivity

The polymer must be a thermoplastic to be able to be melted, reshaped, and then filled with the metal powder to make it electrically conductive to serve as the anode during the reaction. The polymer chosen must also be chemically resistant since the beam will be submerged in 0 ph, 1 mole sulfuric acid for up to ten hours without being corroded. Research has been done on the properties of potential polymers and metals for the components of the beam, but those properties are subject to change once they are combined. The reason for creating this composite is to have the chemical resistance and elasticity of a polymer and the conductivity of a metal, which are requirements for the success of these experiments.

There are several processes that can be used to make polymers and composites; the pure chemical process is the use of a monomer solution and its specific oxidizing agent to create a rigid polymer. Electrochemically is the application of an electrical current through electrodes placed into the solution containing the monomer, the solvent, and the doping agent. A third process is the use of a sonicator, for this process, the monomer, the solvent, and the metal filler are mixed together and placed in the sonar machine. Ideally the sonar machine will be in a pulsar mode, where it is on for a few seconds and off for a few seconds, the vibrations provided by the sonar machine will uniformly distribute the doping agent in the mixture.

The materials chosen must also have a similar Young's Modulus to the MoS₂ in order to transmit as much strain as possible, though it is very unlikely that a material with the same exact Young's Modulus as MoS₂ exists. There is a detailed chart of all material considered and their traits located in Appendix A.

7.2 Mechanism Design

After narrowing down the mechanism designs to four different designs more detailed CAD drawings were created. With these drawings pros and cons were created for each design which helped narrow the designs down to two. Below are the four designs and the pros and cons for each design.

Balloon System

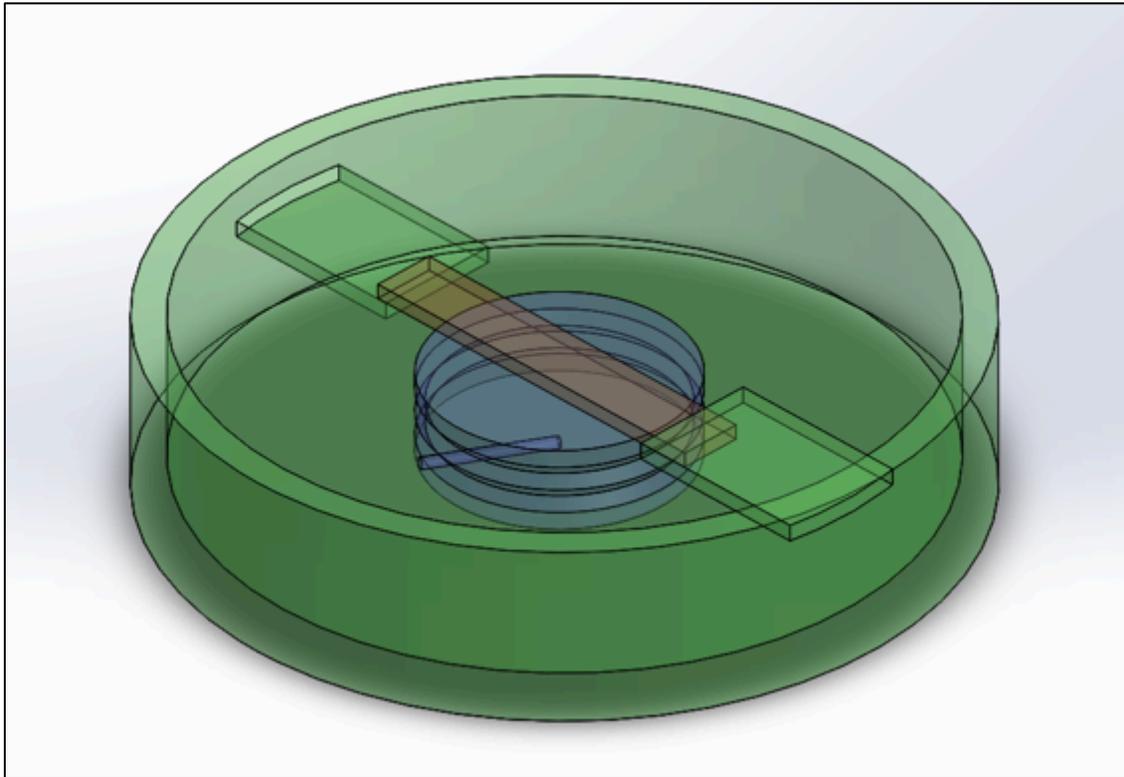


Figure 25: Balloon Design

The Balloon system was created as a design that used a different kind of force to move the beam. Using air pressure to bend the beam is much easier than having to bend the beam using shear force as shown in Figure 25. Air pressure would also be an easy way to measure force induced on the beam and to make sure that the correct amount of strain is being put on the material. The most important part of the balloon design would be that the tube leaving the chamber would not be moving and be able to be statically sealed. All of the other designs created involved a seal which needs to have the ability to move and adjust to the changing surface area of the beam, as well as making sure it is completely sealed and air and liquid tight. This design surpasses all of the other in ease of manufacturing and ability for use.

Though this design has many positives, there are still major flaws. Firstly this design would not result in uniform bending of the beam. When the balloon is inflated it will fill and then touch the beam in one location. Applying force in this one location adds a point force to the beam, which is not uniform bending throughout. Without uniform distribution, the MoS_2 will not

receive the required stress and strain needed for the experiment. In addition to this major obstacle, the material used for the balloon would need to be stronger than the beam to be able to apply the stress. A very strong rubber bladder would be required but it would also take an extreme amount of pressure to inflate, which addresses risk factor to the safety of the experiments.

Four Point Bending- Inside vs. Outside the Chamber Designs

During the initial stages of research it was debated whether the mechanism should be inside or outside the chamber. The idea of having the mechanism outside of the chamber was appealing because of the height constraints of the Raman Spectrometer. The mechanism would have to be small enough to function in the 1.5” space provided if it was inside of the chamber but could branch out on the sides if the mechanism was outside of the chamber. Both inside and outside of the chamber designs were considered

Outside the Chamber Design

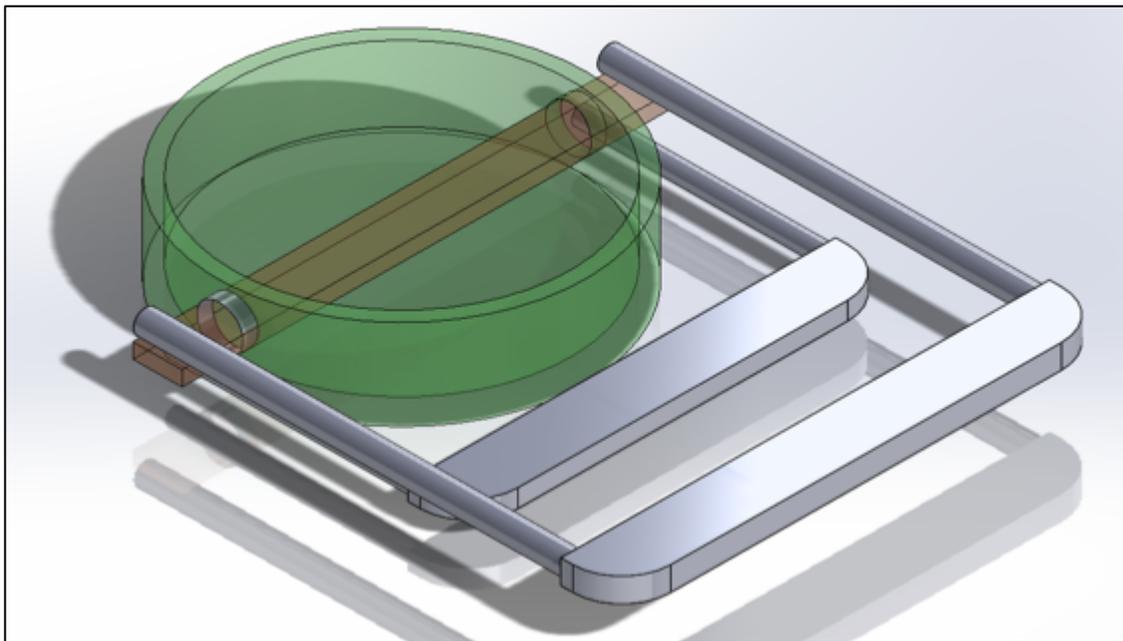


Figure 26: Outside of the Chamber Bending Design

If the mechanism is outside of the chamber, the beam would be longer than the chamber and require custom seals as the surface area of the beam is rectangular and changes with stress and strain. Both design considerations are elaborated on below. Another benefit to this idea was that the mechanism would not have contact with the acid that is inside of the chamber. Since the mechanism causes strain on the part of the beam outside of the chamber, there would be a very small change that it would have contact with the acid during the experiment.

For this design the mechanism would not have to be chemically resistant, though it would be wise to incorporate this property since there are no guarantees there will be no contact. This also helps with the mechanisms durability and lifespan. The less amount of corrosion due to acid

the longer the mechanism could last. Figure 26 shows the initial design of the outside the chamber four point bending method

Inside of the Chamber Design

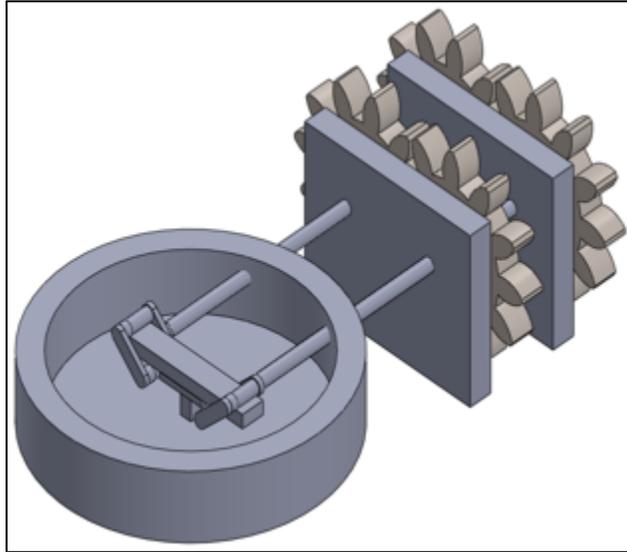


Figure 27: First Iteration of the Inside the Chamber Rotating System

The idea for the inside of the chamber design was developed parallel to the outside of the chamber four point bending idea. These two designs were made up of the same basic design but the inside the chamber design took up a much smaller area. The outside the chamber idea needed to have a much longer beam so the beam would be longer than the diameter of the chamber in order for the mechanism to have enough space to apply strain. For the inside the chamber idea, the pressure points would only be inches apart requiring a much smaller length beam.

With the inside of the chamber idea, the chamber would not have to be sealed to the moving beam. For the beam to come out of the chamber, custom seals would have to be created to fit the rectangular shape of the beam and ensure no acid could seep out of the chamber. The inside the chamber design would only require the mechanism to leave the chamber, which could be made to a shape easier to seal.

The design is simple and contains very few parts. It relies on a simple gear system responsible for one dimension of motion. In addition, this design fits many of the requirements that were set for an excellent design: tension and compression can both be applied and there would only be two seals required where the rods penetrate the chamber. The design was developed further with a CAD model in order to better visualize the motion shown in Figure 27. This is a rough draft of the model without proper dimensions. The gears shown are not the proper size but had the correct orientation for this stage of the development. This design also includes a small platform for the beam to be placed on. As the mechanism rotates, the rods lose contact with the beam. The distance between these rods is much greater than the height of the beam

making it necessary to include a way to balance the beam. The distance between the two rods ensured that both tension and compression can be produced. The mechanism only has holes on one side of the chamber. This is important to note since there would only be two places that require seals.

Pulley System

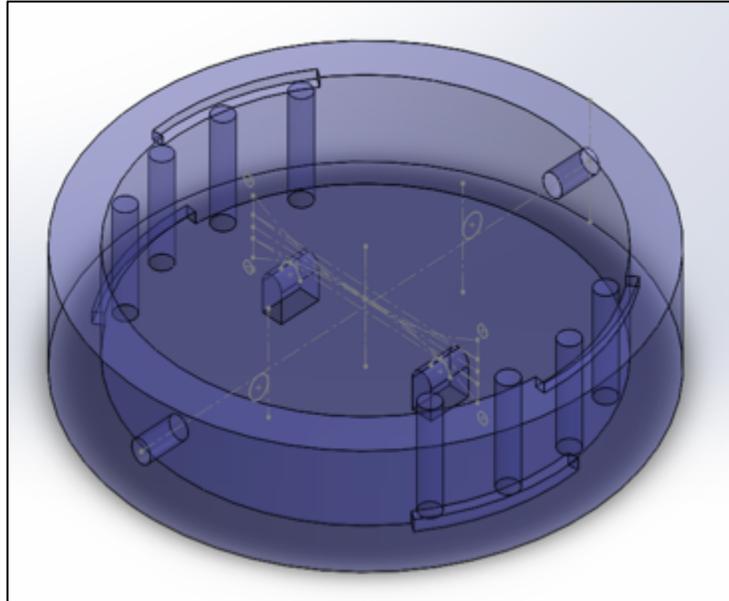


Figure 28: Pulley Design

The above figure depicts the initial pulley design. The pulley design applies force in a four point bending motion. The sketch on the front plane depicts the beam placement and its elastically deformed shape. This was the starting point of the design for clearance checks within the chamber and pulley placement. The pulleys are all of the cylindrical extrusions and circle sketch elements. The cable will be connected to the ends of the beam and wrap around these pulleys going towards the hole in the wall of the chamber. The set of cables that apply tension to the beam will run towards one hole while the second set run towards the other hole. At this stage, the design is purely theoretical, lacking any insight for fabrication.

Final Decision Matrix

With two ideas chosen more detailed designs were created, the pulley design and the pure bending design. These two were chosen because of their more realistic ability to fabricate and succeed than the other designs. After the two designs were chosen more detailed versions of these designs were created.

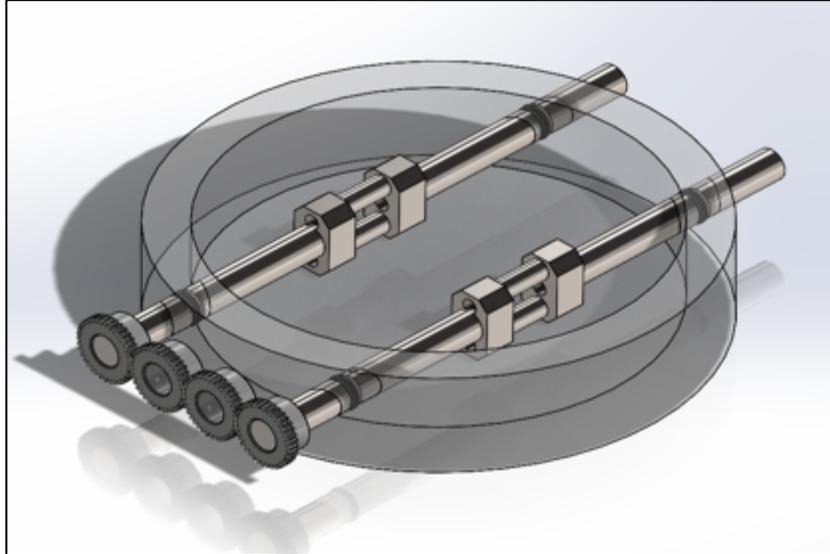


Figure 29: Second Iteration of the Rotating System

The second iteration of the pure bending rotating system is shown in Figure 30 of the rotating system is much more sophisticated than its predecessor. This design has the mechanism going through the entire chamber for more support and a stronger hold on the beam. This design removes the platform for the beam and makes the hold on the beam be the exact width of the beam so that there is no free movement. This changes the type of bending in this design from four point bending to pure bending since there is constant contact on singular points on the beam. The gear orientation changed on this design as well to make it more practical.

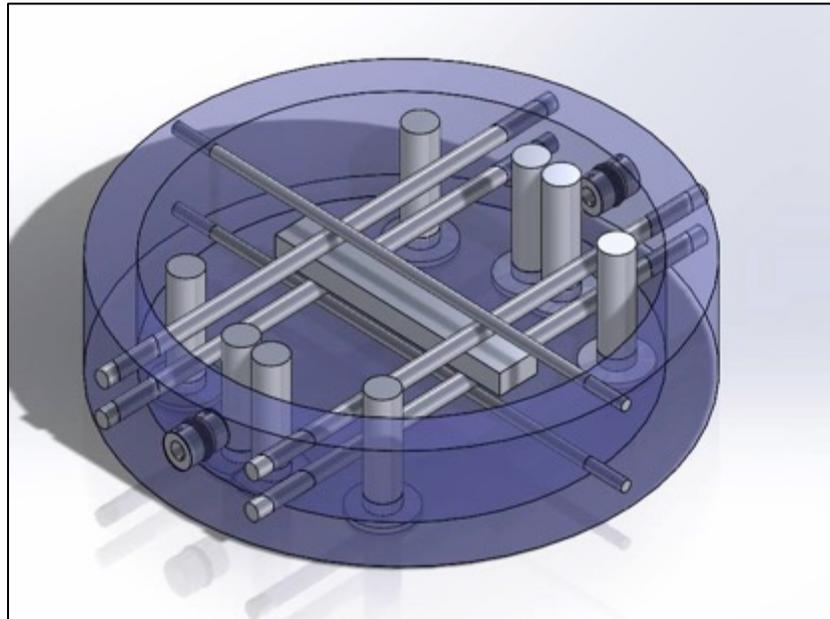


Figure 30: Second Iteration of Pulley Design

The second design that was chosen for further development was the Pulley Design. This design underwent a magnitude of changes for the second iteration. The first noticeable difference is the rods that exit the chamber. These rods represent the pulleys for the cable. A second modification is the small rod that lies inside of the holes in the chamber wall. This is a piston sealed rod that makes the mechanism suitable for cable isolation. The short rod is able to slide while being dynamically sealed. A cable is then attached to the rod from the outside and rigidly connected to the controls.

Fabrication of this design seems realistic, but difficult. Damage to the pulleys is a concern; however, the cables will be moving over small distances (approx. 0.5”) at a slow rate. The chamber will have many holes that need to be sealed. Constraining the cable will also prove to be difficult as the pulleys alone will not be enough to constrain the cable to its desired path.

With two fully developed design ideas, it was time to pick a one to move forward with as the final design. In order to select the best design, the team created a decision matrix with seven design parameters. Each design received a score on a scale of one to three, with three being excellent and a one being subpar. Figure 31 shows the scores that each design received followed by a description of each design parameter.

Design	Safety	Ease of Use	Number of Parts	Replication	Durability	Precision	Manufacturability	Total
Pulley System	2	1	1	1	2	2	1	10
Rotating Points	3	2	3	3	3	2	2	18

Figure 31: Final Design Matrix

Safety

For this requirement the main consideration was what would happen if the mechanism was to break or if the mechanism was put to the extreme. When the pulley design would be pushed to the breaking point the wire could break and then hit other parts of the chamber or injure the person working the device. This could cause harm to the person working the device or damage the chamber so that it’s not fixable. For the rotating design when pushed to the breaking point the beam could break in half or lose contact with the beam. These consequences seem less harmful to the person working the device, for the damage would be kept to the inside of the chamber. For this design the Pulley design received a 2 and the Rotating system received a 3.

Ease of Use

For this category it was important to consider how quickly someone can learn how to properly use the device and be able to replicate the experiment. The quicker a person can figure out how to use the device, the easier it is to use and the easier it is to teach others how to use it. For the Pulley design the user would have to create beams for the experiment very carefully. Holes would have to be drilled into the beam in very precise locations and if these holes were off by even millimeters it would ruin the whole experiment. Also the user would have to attach the beam to the wire in a specific way to make the experiment work. For the Rotating design the beam does not have to be modified at all. Placing the beam inside the chamber will be a small

challenge to make sure that the beam fits in the tight grips for the beam. For these reasons the Pulley design received a 1 and the rotating design received a 2.

Number of Parts

This category was to consider the number of parts that would be in each system for how many parts would need to be assembled and what could potentially break or need to be replaced in the future. The pulley system consists of a wire, bars to put across the chamber, pulleys, gear system, piston system for seal. The rotating system has a gear system, bars to go in and around the beam to cause the bending, O ring for seal. Based on the amount of parts the Pulley system received a 1 and the Rotating system received a 3.

Replication

The ability to replicate the experiments done with this device is very important to the design of the project. For both of these designs the beam is the part that will need to be able to be replicated most. For the pulley system the ability to recreate bar, means there would have to be holes drilled into both side, and some way to connect the pulley to the bar. This is a complex design that will be difficult to replicate and make sure that all the beams are the same. For the rotating design the beams only requirement is to be a simple rectangular bar that will fit between the clamps. These are the reasons that the Pulley design received a 1 and the rotating design received a 3 for replication.

Durability

The durability category for the decision matrix is to decide which design would do best over repeated use and wear. The pulley system has multiple pulleys and bars that these pulleys will be located on, all which will have force directly applied to them in singular locations and will repeatedly move during the experiments. These tests will affect the durability of these parts, though with the material they are made of will result in the amount of wear on the material. The rotating design has few parts that will be rubbing or causing wear on other parts, increasing the durability. For durability the Pulley system received a 2 and the rotating system received a 3.

Precision

The devices will need to be used for precise experiments so the precision of the mechanism must be exact. Both of the designs proposed have similar gear boxes which results in the precision of them is the same The problem would be the ability to get the devices to stop at the exact location where the gears are at the values wanted. This issue is the reason both systems received a 2.

Manufacturability

The last category that is important to consider would be the manufacturability of the systems. The pulley system required a lot of changes and additions to the chamber, which makes

it difficult to manufacture, and in addition everything would have to be in specific locations adding to the difficulty of manufacturing. The rotating system requires exact length and location of gears and the gears also have to be extremely precise making the manufacturability difficult. Also the location of the clamps for the bar have to be close together so that the beam will be able to be held snugly, but so that the beam will also be able to be removed and replaced. The Pulley system received a 1 for manufacturability and the Rotating design received a 2.

Final Design Decision

After scoring the different designs in the categories believed to be the most important characteristics, the Rotating System was chosen for the final design. This design received either an equal or better score than the Pulley System in every category. Figure 32 shows the last version of the Rotating System design that was used in the decision process.

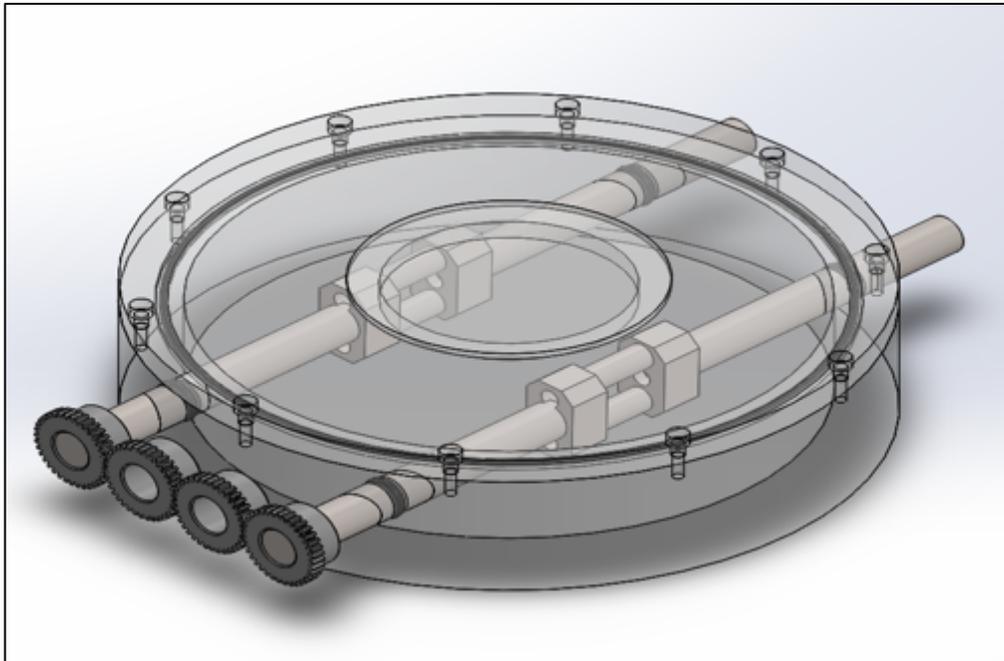


Figure 32: Rotating System with Lid

8.0 Final Design and Mechanical Analysis

8.1 Designs

There are five major components to this design; the arm subassemblies, chamber, lid, input gear train and the baseplate. The arm subassemblies transfers torque to the beam and is comprised of three parts, the shaft, coupler, and fixture to hold the beam. Torque applies pure moment loading to the beam. Rectangular prisms with square cross-sections were used to apply the same torque through two planes clamping onto the beam. The fixturing in this case is comparable to a vice. Using the original cylindrical fixturing concept would be analogous to

using a vice whose teeth were cylindrical. The clamping force would be concentrated on a line, which would not provide a reaction moment to prevent rotation about that line.

The chamber can be separated into two parts, the lid and chamber body. These two parts are geometrically complicated and require CNC machining for accurate manufacturing of critical dimensions. The chamber serves as the main body of the chemical cell and also acts as a “bearing” mount for the shafts of the mechanism arm sub-assembly. The lid provides a means of sealing the chemical cell while accommodating a quartz window and custom fittings for the different chemical apparatuses necessary to conduct and monitor the chemical reaction.

The input gear train is vital for adjusting the applied strain in small increments. The gears are mounted to the shafts through setscrews. Bug testing of the entire device will determine whether or not keyways are required. The shafts are press fit into bearings for proper mounting and meshing of gears and shafts. A means of position locking is required. This will be implemented through a vertical gear rack that can slide into the gear mesh to prevent the gear it is meshed with from rotating.

The base plate provides a common ground for the mounting of all other components. This will be the final piece of the system since its dimensions are dictated by the previously discussed components. The detailed final design is pictured in Figure 33.

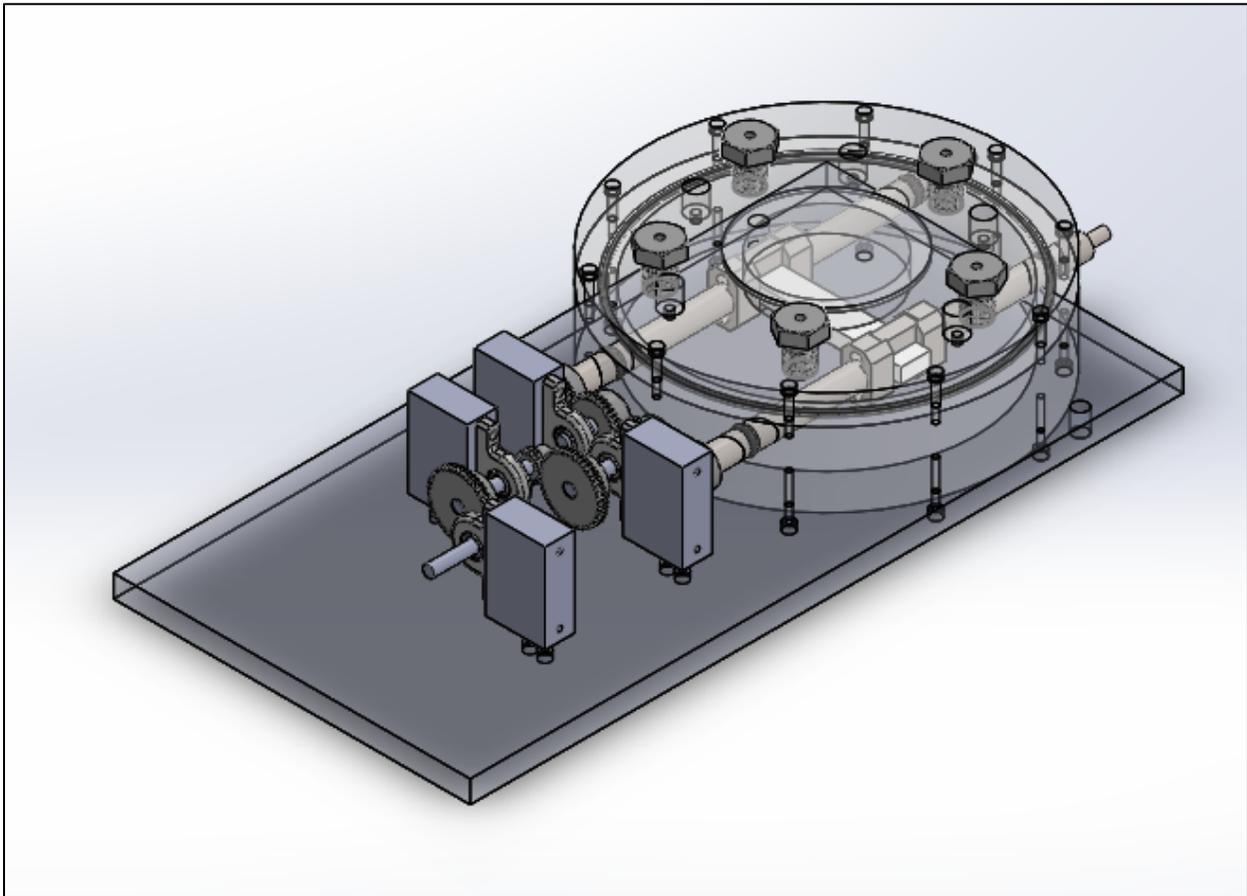


Figure 33: Final Design

8.2 Mathematical Calculations

The main objective of the device is to apply strain to a specimen of MoS₂. Therefore, the starting point for mathematical analysis begins with the physical design of the substrate for the MoS₂. Pure moment is applied to a beam with symmetric conditions such that the bending moment as a function of beam length is constant throughout the section of desired MoS₂ placement. The free body diagram, in Figure 34 depicts the loading scenario. This creates a moment controlled strain device.



Figure 34: FBD of Beam in Pure Bending

Two things are now in consideration at this point, Input torque for desired strain and max beam deflection. The Input torque is necessary to determine states of stress within the force transmission components, while the maximum beam deflection is necessary to determine how much clearance is required within the chamber to avoid bottoming out. To figure out the strain that would be put on the beam preliminary calculations were done. Calculations for both pure bending and four point bending were done, for pure bending is dependent on the beam being exact size that the mechanism is, and if it is not the beam will be under four point bending. A beam in four point bending is analyzed first. Then, a beam in pure bending is analyzed and verified against the four point bending model.

Analysis for a Beam in 4 - Point Bending

Input

$F := 22$ [lbs] $L := 3$ [in] $c := 0.5$ [in]
 $E := 95000$ [psi] $a := 0.5$ [in] $d := 0.21$ [in]
 $b := L - a$ [in] $I_x := \frac{c \cdot d^3}{12}$ [in⁴]
 $S(x, z) := \text{if}(x \geq z, 1, 0)$ $x := 0, 0.001 \cdot L .. 0.999 \cdot L$ [in]
 Find Reaction Forces $t := 2.787 \cdot 10^{-8}$ [in]

$$\Sigma F = -2 \cdot F + R_1 + R_2 = 0$$

$$\Sigma M = -L \cdot F + a \cdot R_1 + (L - a) \cdot R_2 = 0$$

We can then solve the system of equations with Linear Algebra

$$A := \begin{pmatrix} 1 & 1 \\ a & b \end{pmatrix} \quad B := \begin{pmatrix} 2 \cdot F \\ L \cdot F \end{pmatrix}$$

$$R := A^{-1} \cdot B = \begin{pmatrix} 22 \\ 22 \end{pmatrix}$$

$$R_1 := R_0 = 22 \quad \text{[lbs]}$$

$$R_2 := R_1 = 22 \quad \text{[lbs]}$$

Figure 35: Reaction Forces of 4 Point Bending

This is the first section of the Mathcad code that establishes variable declarations and static force calculations. Figure 35 states that, under symmetric conditions, the reaction forces equal the input force.

F is the input force.

L is the length of the beam.

a and **b** are the locations of the reaction forces.

c and **d** are the cross sectional dimensions of the beam.

E is the young's modulus of the material.

x is the discretization of the domain in steps of one thousandth's of the beam length.

t is the thickness of the monolayer MoS₂.

I_x is the moment of inertia of the beams cross section.

Loading Equation

$$q(x) := -F \cdot S(x, 0) \cdot (x - 0)^{-1} + R1 \cdot S(x, a) \cdot (x - a)^{-1} + R2 \cdot S(x, b) \cdot (x - b)^{-1} - F \cdot S(x, L) \cdot (x - L)^{-1}$$

Shear Equation

$$V(x) := -F \cdot S(x, 0) \cdot (x - 0)^0 + R1 \cdot S(x, a) \cdot (x - a)^0 + R2 \cdot S(x, b) \cdot (x - b)^0 - F \cdot S(x, L) \cdot (x - L)^0$$

Moment Equation

$$M(x) := -F \cdot S(x, 0) \cdot (x - 0)^1 + R1 \cdot S(x, a) \cdot (x - a)^1 + R2 \cdot S(x, b) \cdot (x - b)^1 - F \cdot S(x, L) \cdot (x - L)^1$$

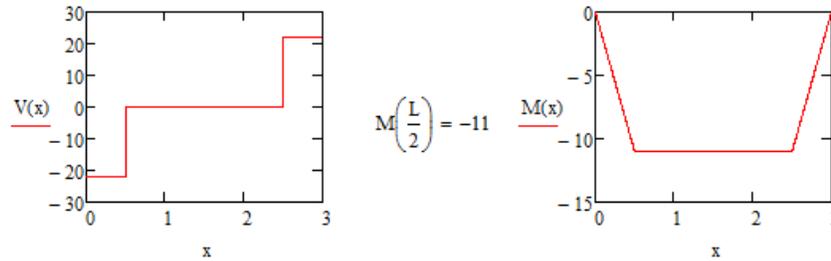
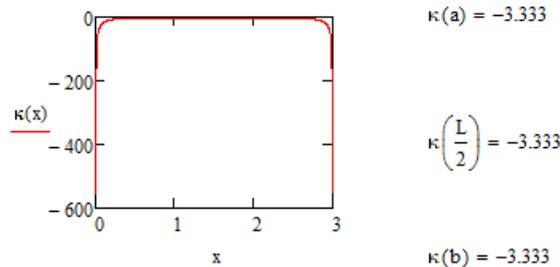


Figure 36: Shear and Moment Diagrams

The loading function, $q(x)$, is a mathematical statement that depicts all of the loading conditions on the beam. For this case, only point forces are acting on the beam indicated by the exponent of -1. Moments and distributed loads have different exponent values. Using the reaction forces, the shear $V(x)$ and Moment $M(x)$ diagrams are constructed in Figure 36. The diagrams are derived through the integration of $q(x)$. The constants of integration, R1 and R2, have been predetermined in this case from the static analysis. This obtains a constant moment throughout the middle section of the beam, demonstrating that the strain on the surface of the beam is uniform from points $a - b$. This fulfills the requirement of uniform strain for the MoS₂.

The curvature is related to the moment equation through the following relation:

$$\kappa(x) := \left(\frac{M(x)}{E \cdot I_x} \right)^{-1}$$



The strain of the sample can then be calculated through $\epsilon = t / R$

$$\epsilon_b := \frac{\left(t + \frac{d}{2} \right)}{\kappa \left(\frac{L}{2} \right)} = 0.032 \quad \text{Limit this to 3\% (0.03) strain}$$

Figure 37: Curvature and Strain of the Beam

Because the moment between **a** and **b** is constant, the curvature of the beam is also constant, creating a simple relation for the strain at the surface of the beam.

Above, Figure 37 depicts the curvature $k(x)$, of the beam as a function of x and the strain at the surface of the beam. The input force **F** is given arbitrary values until the beam strain ϵb reaches a value of 0.03 (3%) strain. Once this value is reached, the input torque and beam deflections can be calculated. The torque in this case is simply the $M(x)$ value in the region from **a** – **b**. This value is about 11 in-lbs.

To obtain equations for the slope and deflection of the beam we must obtain integration constants using the constraints $y(a) = 0$; $y(b) = 0$. This will yield a system of equations that can be solved with Linear algebra.

$$D := \begin{pmatrix} a & 1 \\ b & 1 \end{pmatrix} \quad G := \begin{bmatrix} \frac{F \cdot a^3}{6} \\ \frac{F \cdot b^3}{6} + \frac{-R1}{6} \cdot (b - a)^3 \end{bmatrix}$$

$$C := D^{-1} \cdot G = \begin{pmatrix} 13.75 \\ -6.417 \end{pmatrix}$$

$$C1 := C_0$$

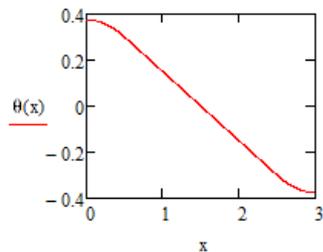
$$C2 := C_1 \quad +$$

Slope Equation

$$\theta(x) := \frac{1}{E \cdot Ix} \left[\frac{-F}{2} \cdot S(x,0) \cdot (x-0)^2 + \frac{R1}{2} \cdot S(x,a) \cdot (x-a)^2 + \frac{R2}{2} \cdot S(x,b) \cdot (x-b)^2 - \frac{F}{2} \cdot S(x,L) \cdot (x-L)^2 \dots \right] + C1$$

Deflection Equation

$$y(x) := \frac{1}{E \cdot Ix} \left[\frac{-F}{6} \cdot S(x,0) \cdot (x-0)^3 + \frac{R1}{6} \cdot S(x,a) \cdot (x-a)^3 + \frac{R2}{6} \cdot S(x,b) \cdot (x-b)^3 - \frac{F}{6} \cdot S(x,L) \cdot (x-L)^3 \dots \right] + C1 \cdot x + C2$$

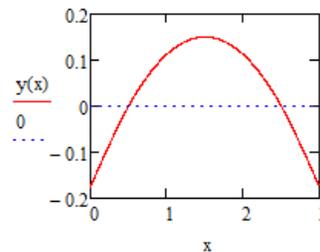


Slope of the beam in radians.

$$\theta(0) = 0.375 \quad \theta(L) = -0.375$$

$$\theta\left(\frac{a}{2}\right) = 0.356 \quad \theta\left(\frac{L-b}{2}\right) = 0.356$$

$$\theta(a) = 0.3 \quad \theta(b) = -0.3$$



$$y\left(\frac{L}{2}\right) = 0.15 \quad \text{+/- Displacement of specimen to the lens in inches.}$$

Figure 38: Slope and Deflection Diagrams

Figure 38 depicts the slope $\theta(x)$ and deflection $y(x)$ in radians and inches respectively. These equations are formulated through integrating $q(x)$. The resulting constants of integration are $C1$ and $C2$, determined through the use of linear algebra. The maximum deflection of the

beam at midspan is 0.15 in. The total deflection of the midspan (peak to peak) is 0.3 in. This will cause the Raman microscope to become unfocused once strain is applied. Therefore, the Raman microscope should be refocused at the desired level of strain.

Input Rotation to strain calculation

Input

$E := 95000$ [psi] $\varphi := 0, 0.001 \dots \frac{\pi}{4}$ Input Rotation Variable

$c := 0.5$ [in]

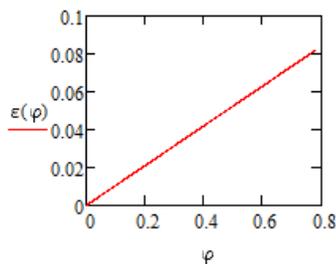
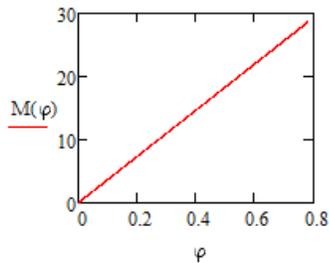
$d := 0.21$ [in]

$I_x := \frac{c \cdot d^3}{12}$ [in⁴]

$M(\varphi) := \varphi \cdot E \cdot I_x$ Moment Calculated from Input Slope

$R(\varphi) := \frac{M(\varphi)}{E \cdot I_x}$ Radius of curvature = φ

$\varepsilon(\varphi) := \frac{d}{2} \cdot \varphi$ Bending Strain



$\eta := \frac{0.032 \cdot 2}{d} = 0.305$ [rads]

$\eta \cdot \frac{180}{\pi} = 17.462$ [degrees]

$M(\eta) = 11.172$ [in-lbs]

Bending strain is linear with respect to input rotation. This is an expected and desirable result.

Figure 39: Beam in Pure Bending Mathcad Code

Figure 39 contains the entire Mathcad code for the beam in pure bending. The moment diagram $M(\varphi)$ is a linear function of input rotation φ in radians. This relation can be derived from the slope formula in Figure 38, which is a well-known expression from continuum mechanics, assuming $\theta(x) \ll 1$ [rad]. This assumption holds true for small distances away from the center of the beam since $\theta(L/2) = 0$. Therefore, it is safe to calculate the strain of the MoS₂ specimen in this way.

The curvature, ρ , is defined as $\varphi = \rho^{-1}$ and $\varphi = 0$, $\rho = \infty$, which correlates to the curvature of a line. All of these physical conditions are represented within the Moment and strain diagrams, allowing for the direct correlation of strain to input rotation. It was determined that 18

degrees of rotation yields 3.2% strain and requires 11.17 [in-lbs] of torque. All of these values match the solution of the beam in 4 point bending. This is the theoretical relation that will be experimentally verified with a strain gauge and potentiometer.

9.0 Manufacturing and Assembly of Final Design

9.1 Beam Manufacturing

To create the beam a composite material that would be electrically conductive and be able to transfer the amount of strain wanted needed to be created. There are multiple processes that have had success in creating similar polymers and composites. One of the most common methods for making polymers is the pure chemical process. This process utilizes a monomer solution and its specific oxidizing agent to create a rigid polymer. This process has been proven to be time efficient while still producing a well-composed polymer. Another method that is regularly used is electrochemically. This process is the application of an electrical current through electrodes placed into the solution containing the monomer, solvent and doping agent. The electric current uniformly distributes the doping agent in the solution.¹⁴ A third option to uniformly distribute the doping agent is the use of an ultrasonic machine. In this process the monomer and the solution are combined with the doping agent and placed inside the ultrasonic machine. The high frequency agitation should uniformly distribute the dopant into the solution. Given the equipment that is available in the lab, this method is the most suitable for this experiment.

Once the method was determined, materials needed to be selected to conduct the experiment. The first two materials used were High Impact Polystyrene (HIPS) and copper. In order to ensure a proper mix, 3g of HIPS was dissolved in pure acetone before 1g of copper was added to the mixture. This mix in the beam gave 33% of copper in the composite.

After being thoroughly mixed, the solution was placed on a high sonar machine for ten minutes to ensure uniform distribution of copper in the plastic. Next the composite was placed in the mold in order to shape and dry.

After forty-eight hours had passed, the mold was opened to reveal that the composite had not completely dried. The mold was opened and left to dry. Unfortunately the surface did dry but formed many air bubbles in the middle of the beam as shown in Figure 40. Even though this sample was ruined by shape and form it was still tested to see the electrical conductivity using a multimeter. This test yielded a resistance far too great to conduct any kind of electricity. The beam was also broken due to hand tests of elasticity. This failure presented a base for further combinations of materials.

¹⁴ Balint, R., Cassidy, N. J., & Cartmell, S. H. (2014). Conductive polymers: Towards a smart biomaterial for tissue. *Acta Biomaterialia*, 10, 2341-2353. Retrieved November/December, 2015.

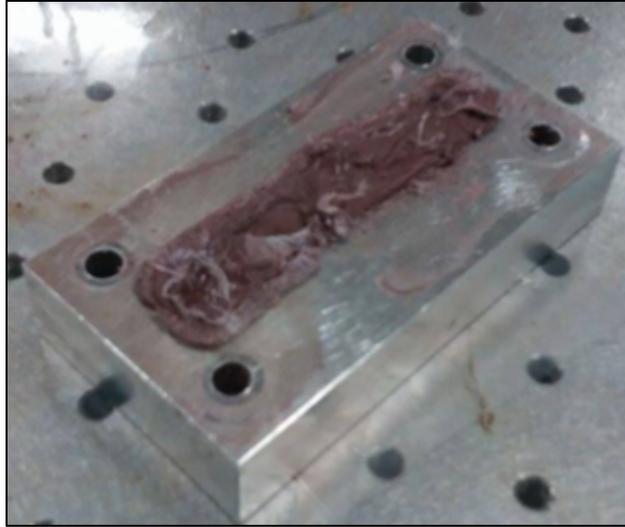


Figure 40: First Trial for the Beam Made from Polystyrene

In the second attempt at creating a suitable beam, the acetone was heated in order to melt the polymer quicker. This mixture also used HIPS and copper but this time used 0.0105 pounds and 0.042 pounds respectively. This time there was a greater weight percentage of copper was used in order to see how it would affect the electrical conductivity of the beam.

Though during the drying process, air bubbles formed in the middle of the beam again, and it still could not conduct any electricity even with almost 50 % of copper. With this weight percentage, it is unlikely that the resulting beam would still retain any of the plastic properties necessary for a successful beam. After curing the composite, the beam was taken out of the mold. Pictures of the top and bottom of the beam are shown in Figure 41 and Figure 42. The pockets of air that had formed in composites also make the resulting beam more brittle and able to break with little pressure this is shown in Figure 43. It was discovered that the bottom was highly conductive. All of the copper used had sunk to the bottom of the mold. This was also not usable for this experiment. With these failures in mind, research was done into alternative methods.



Figure 41: Top of the Second Attempt for the Beam



Figure 42: Bottom of the Second Attempt of the Beam



Figure 43: Inside of the Mold Created Beam

After some research a process called spin coating seemed to match the needs of a successful beam. In this process, the solid polymer is placed inside of a spin coating machine. The polymer is secured through a vacuum while it rotates and the solution spreads out on the top surface. The excess solution is removed as the object rotates. The faster the piece is rotated, the thinner the layer will be. The thickness of the layer is key since an improper thickness could mean the beam is nonconductive and brittle.

Below are descriptions of the different trials of spin coating that were performed. These descriptions include the material and the amount of the material that was used. Also they include the procedure that was followed during the experiment.

Trial One



Figure 44: Trial One of Spin Coating of the Beam

Materials

- 1.1 g of HIP pellets
- 0.35 g of Cu flakes
- Acetone

Preparation

- The PS was melted in acetone and mixed with Cu, then it was placed in the (sonar machine) to have a uniform distribution of Cu inside of the PS.
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programed to spin at 2000 RPM for 40 seconds.
- The paste did not spread evenly on top of the beam, we believe it is because it was too viscous to spin coat. The solution would be to increase the speed, but the vacuum was too weak to have a speed above 2000 RPM with the beam dimensions and weight.

Trial Two

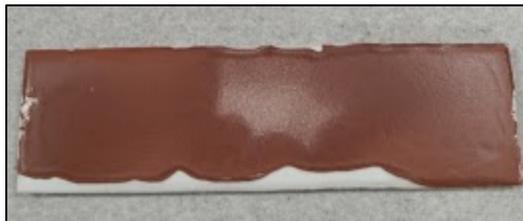


Figure 45: Trial Two for Spin Coating the Beam

Materials

- 3g of PMMA
- 1g of Cu flakes
- Acetone

Preparation

- The PMMA and Cu were mixed together and place in the sonar machine to mix them
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programed to spin at 3000 RPM for 40 seconds.
- The speed was to great for the viscosity of the solution and it spin most of the solution out of the beam

Trial Three



Figure 46: Trial Three for Spin Coating the Beam

Materials

- 3g of PMMA
- 1g of Cu flakes
- Acetone

Preparation,

- The PMMA and Cu were mixed together and placed in the sonar machine to mix them
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programmed to spin at 2000 RPM for 40 seconds.
- The speed was too great for the viscosity of the solution and it spun most of the solution out of the beam

Trial Four

Materials

- 2.8 ml of PMMA
- 1 g of Cu flakes
- Acetone

Preparation

- The PMMA and Cu were mixed together and placed in the sonar machine to mix them
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programmed to spin at 1000 RPM for 40 seconds.
- The speed seems low, and it gives a thick layer of material to the beam, one layer is not conductive, but with ~33% of Cu it should be conductive.
- The second layer of solution is still not conductive.
- The third layer is also not conductive.

To see why the beam was not conducting electricity, some SEM images were taken. The figure below is a SEM image of the beam slightly tilted.

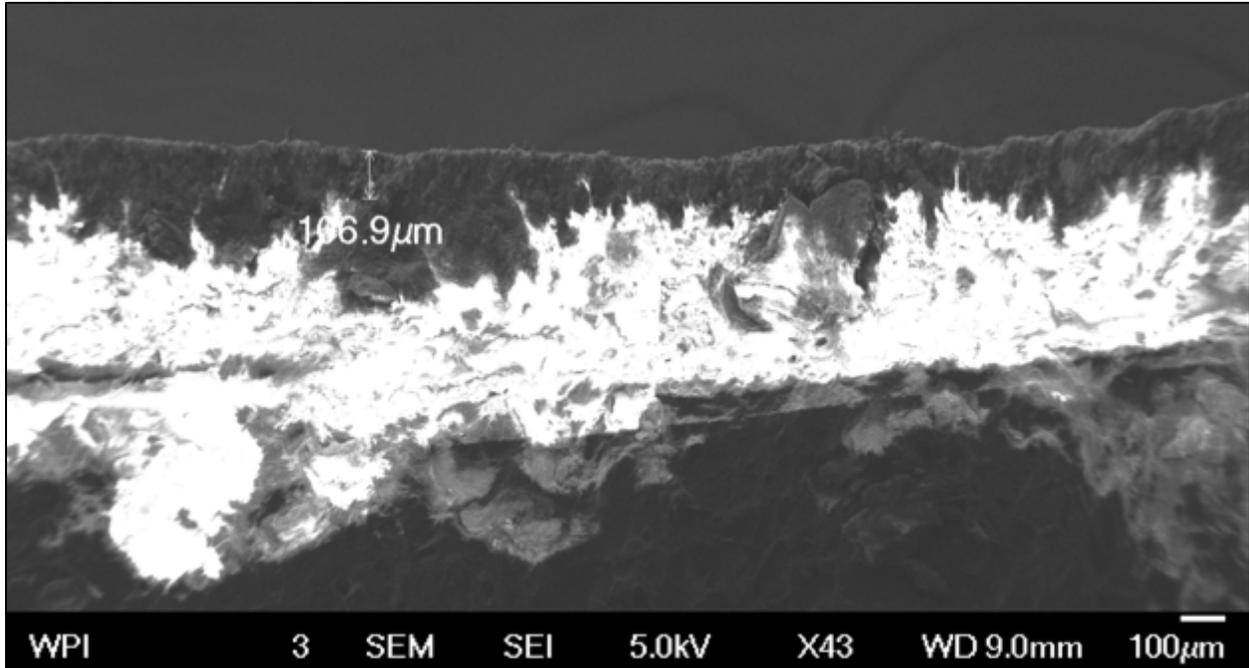


Figure 47: SEM image of tilted beam

The scale is reading 106.9 microns, it is an extremely thick layer for a spin coat, and because a SEM can be used, it is conductive. A picture of the surface of the beam was also taken with the microscope to see what it looks like.

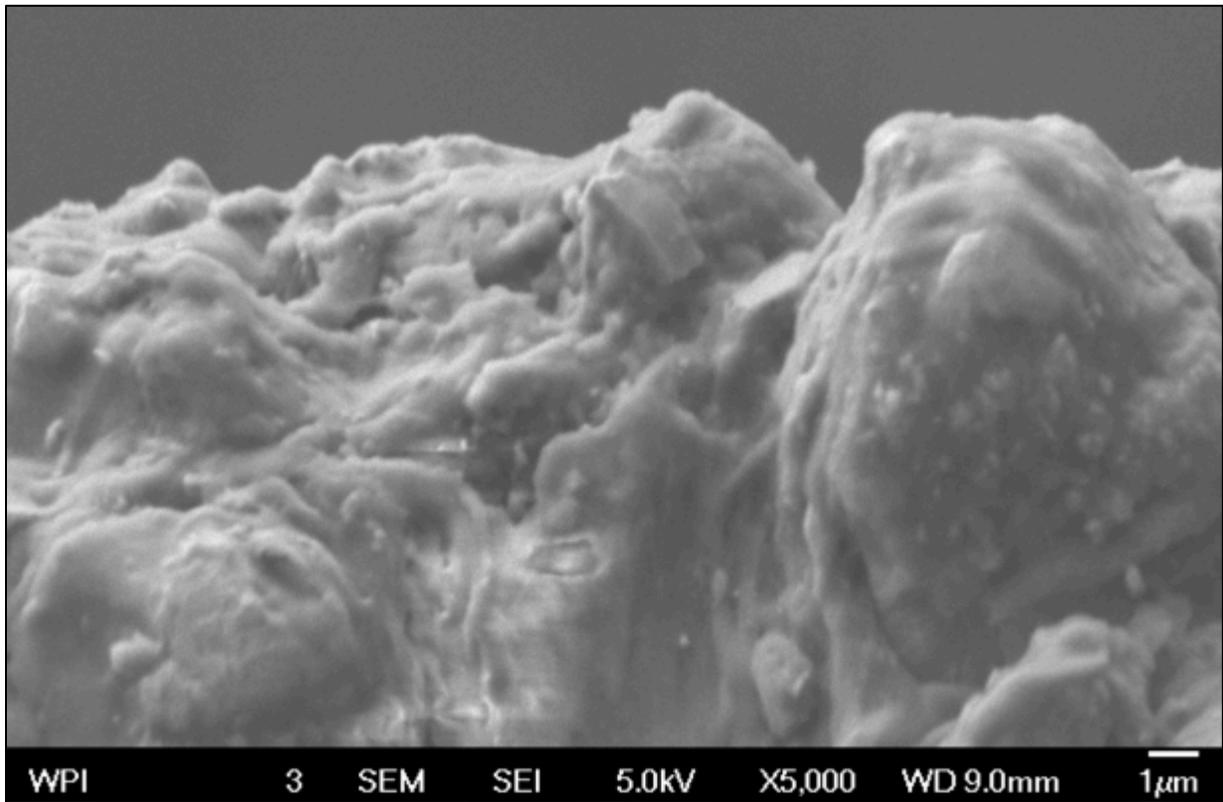


Figure 48: SEM Beam Surface

As seen in the surface of the beam, the copper is under a layer of PMMA. With that in mind, it was suggested that polishing the surface of the beam could expose the copper flakes and make it conductive. The figure below is a picture of the polished beam.



Figure 49: Polished Beam

The figure shows a very shiny beam, it looks like it is pure copper, but it is still not conductive. The best guess is that the copper flakes are not dispersing in the solution and they are not interacting with each other enough to conduct electricity,

Trial Five

For the next trials, only about 5-6% of PMMA was used, since one of the theories was that the PMMA was too viscous to by itself for this coat, and was giving a coat that was too thick. It was decided to try 5 wt% of PMMA and Anisole for the mixture.



Figure 50: First Layer of Trial Five

Materials

- 0.18 ml of PMMA
- 1 g of Cu flakes
- 2.8 ml of Anisole

Preparation

- The PMMA, Anisole, and Cu were mixed together and place in the sonar machine to mix them
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programed to spin at 2000 RPM for 40 seconds.

- The speed seems high, since it is throwing almost all of the mixture out of the beam.
- Try a second layer with a slower speed.



Figure 51: Second Layer of Trial Five

The second layer was coated with a speed of 1000 rpm for 40 seconds and had the same composition as the first layer. It had a more uniform distribution than the first layer, but it seems that it is creating random defects on the surface of the coat. It was still not conductive. A third layer was added to the beam. The figure below shows the beam with the third layer.

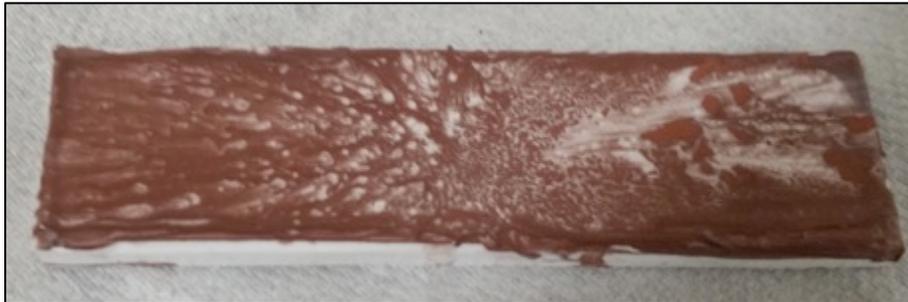


Figure 52: Third Layer of Trial Five

The same composition and process as the second layer were used; the mixture is opening random holes in the coat as it is spun out of the beam. This layer is even showing a non-uniform distribution of copper in certain spots. The result is not conductive.

Trial Six

For the next layer Triton X was used. Triton X was added to the copper and ethanol to make the copper particles hydrophilic, in the hope to get a better dispersion in the mixture.

Materials

- 0.18 ml of PMMA
- 1 g of Cu flakes
- 2.8 ml of Ethanol
- a few drops of Triton X

Preparation

- The PMMA and 1 ml of Ethanol were mixed together and placed in the shear mixer for about 5 minutes

- Copper, a few drops of Triton X, and 1.8 ml of Ethanol were mixed together and place in the sonar machine for about 5 minutes
- After this time, both mixtures were combined and placed for shear mix for another 2 minutes
- The beam was placed in the spin coater and the mix was spread on top of it.
- The spin coater was programed to spin at 1000 RPM for 40 seconds.



Figure 53: First Layer of Trial Six

- By inspection, 1000 RPM was too fast, the speed was decreased to 500 RPM for 40 seconds when applying the next layer.



Figure 54: Second Layer of Trail Six

- A third layer, and a fourth layer were added with the same composition.



Figure 55: Third Layer of Trial Six

The fourth layer was conductive. The multimeter read around 128 k(ohms) in certain places. The average conductivity was tested using two stripes of copper tape about a millimeter apart, which did not show any conductivity.

After many trials with different compositions, processes, and amount of layers the problem of a non-uniform distribution of copper in the mix persisted. One reason for its occurrence could be the size of the copper flakes, about 3-5 microns. They could be too heavy

for the solution and be sinking to the bottom, causing the non-uniform distribution. Other reason could be improper shear mixing since there was only magnetic stirrers for that. Therefore, it was decided to stop trying to get a conductive layer on the beam and focus on testing the mechanical system.

9.2 Mechanism Manufacturing

When creating the mechanism, the materials needed to be chemically resistant but would also be strong enough to bend the beam to the required stress and strain. When considering the material of the beam to be High Impact Polystyrene the mechanism must be made sure to be strong enough to bend this. Since HIPS is a fairly stiff material, metals like aluminum were discarded because it will become deformed when trying to bend the beam. A stronger material was required in order for this device to function. After conducting research on durable metals that are chemically resistant as well as easily to machine and obtainable, type two titanium was chosen. Type two titanium is a material that is extremely strong, but also can be manufactured easier than type one titanium. Titanium is chemically resistant and is very durable for long-term experimentation. This material could be purchased from suppliers such as McMaster Carr in different shapes and quantities, which is ideal for the uniquely shaped parts the design requires.

Due to the simplicity of the parts, the majorities could be manually machined. This was done in the Higgins Machine Shop on the manual mills and lathe. The final dimensions of each part were chosen for ease of manufacturing to reduce machining time. After consulting machinists, special tooling for titanium is only for shops that mass-produce titanium parts. Tools used for high-speed machining of steel are sufficient for the project's needs. These tools are in abundance in the machine shops on campus and with the help of the lab monitors, feeds and speeds were determined that would create parts with high quality surface finish. For this machining using coolant is absolutely critical for tool life and for avoiding unwanted local heat treatment of the work piece. The HAZ of a titanium part becomes very hard and often times break the current tool, rendering it useless.

The four couplers; shown in Figure 56, are the most complicated parts to machine. These four pieces brings the mechanism together thus making them the most essential pieces to machine. These parts were machined on a CNC mini mill to ensure that all the dimensions were exact.

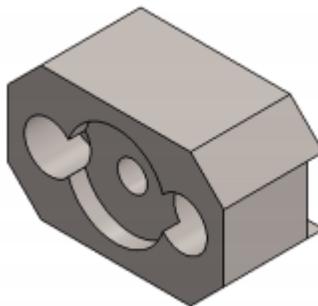


Figure 56: Mechanism Coupler

9.3 Chamber Manufacturing

The chamber was required to be CNC machined in order to have it be suitable for the experiment. The chamber required two different stocks, one for the lid and the other for the chamber body. Both stocks have a diameter of 6 inches enabling the operator to skip an outer diameter operation. Given this diameter, the chamber has enough room to house the beam and the mechanism while they are used in the experiments.

The chamber was required to be CNC machined in order to have it be suitable for the experiment. Utilizing the ESPRIT CAM software, g-codes were created and sent to the HAAS mill to machine. This HAAS mill machine is different from the one used to manufacture the coupler for the mechanism due to the difference in materials. Since the chamber is made out of Teflon, it had to be machined in the Higgins Machine Shop, which handles most of the plastic machining on campus.

Separate ESPRIT programs were created for the chamber body and the lid. Each had a separate stock that was purchased to the proper diameter and thickness. These numbers were chosen in regards to the dimensions of the beam, mechanism and Raman spectrometer. The tools used during machining of both parts were $\frac{3}{8}$ " end mill, $\frac{3}{16}$ " end mill, $\frac{1}{16}$ " end mill. Once CNC machining was completed, each part was cleaned using manual tools.

10.0 Testing of Final Design and Results

10.1 Beam Tests

Tests were performed on the beam to ensure that it meets the requirements. As stated in Project Specification section, the beam must be electrically conductive and stay conductive while under strain. It must also be able to be flexible enough to tolerate the applied strain without plastically deforming.

The first test was measuring the initial conductivity of the beam. This is the first test that was conducted because if the beam is not conductive, then it is not suitable for this project. To test the conductivity, a multimeter made contact with the beam at the opposite ends of the copper spin coat layer. This was to ensure the copper layer is uniform which ensures a uniform distribution of conductivity. Once the beam passes the initial conductivity test, its flexibility must be tested. A pressure gauge is attached to the top of the beam to measure the amount of strain that is applied. The first measurement tested will be at 0% strain, then at 1% and 2% to make sure that no plastic deformation would occur. During each trial the multimeter is used again to measure the conductivity of the beam. This will determine if the beam is still conductive while under strain. These percentages were chosen for testing since they are the percentages that will be used during experimentation.

In order to test the LabVIEW program the theoretical strain of the beam was calculated by clamping one side of it and placing a known weight on the other side.

The equation used was: $\epsilon = \frac{M(x)y}{EI}$

Where $M_{(x)}$ is the moment $M_{(x)} = m \cdot x = (0.807 \text{ lb}) \cdot (3 \text{ in})$

Y is the thickness $y = 0.24 \text{ [in]}$

E is the Young's modulus, $E = 326334.9099 \text{ [psi]}$

I is the moment of inertia, in the case of a rectangular cross-section $I = \frac{1}{12}bh^3 = \frac{1}{12}(1)(0.24)^3$

The equation yields:

$$\epsilon = \frac{M(x)y}{EI}$$

$$\epsilon = \frac{(0.807)(3)(0.24)}{(326334.9099)\left(\frac{1}{12}(1)(0.24^3)\right)}$$

$$\epsilon = 0.00202 = 2.02 \text{ millistrains}$$

The program was reading about 1.91 millistrains.

Therefore, the program is working and reading a value close to the theoretical value.

10.2 Mechanism Tests

Strain tests were conducted using the mechanism to ensure that it is capable of applying the necessary strain. A test beam was created with a strain gauge attached to it. This strain gauge is hooked up to a LabVIEW program, described in APPENDIX B: Beam testing procedure, which will display the amount of strain induced on the beam. This test beam will be used to calibrate the mechanism since the amount of strain per gear revolution can be solved for. This will enable the operator to quickly set the mechanism to the desired increment of applied strain.

Similarly to the beam testing, the mechanism also must be tested for longevity. Once calibrated, the mechanism will be set to 1% then 2% for varying time increments. These increments are 5 minutes, 15 minutes, 15 minutes, 30 minutes and 60 minutes. This final test will ensure that the mechanism is capable of applying different strain levels for an extended period of time.

Test One

The first test of the mechanism was done without the gear train to test the limits of the strain gauge and have initial data for strain vs degrees of rotation. Two vice grips were used to apply force, each attached on separate couplers. A method to measure the angular displacement that the beam was bending was required in order to graph the relationship between the strain and

the degrees of rotation. At this time using an automatic level seemed to be the most efficient method. This tool can be found on an iPhone. The iPhone was held flat to the top of a coupler to read the angle of rotation, as shown in Figure 57.



Figure 57: Test Trial 1

In previous calculations it was determined that the beam needed to be displaced about 4% to be able to strain the beam the necessary 2%. When the beam was put in compression it was quickly discovered that the beam would hit the bottom of the chamber before the 4% strain was reached. The graph from the first test of the beam in compression is shown in Figure 58.

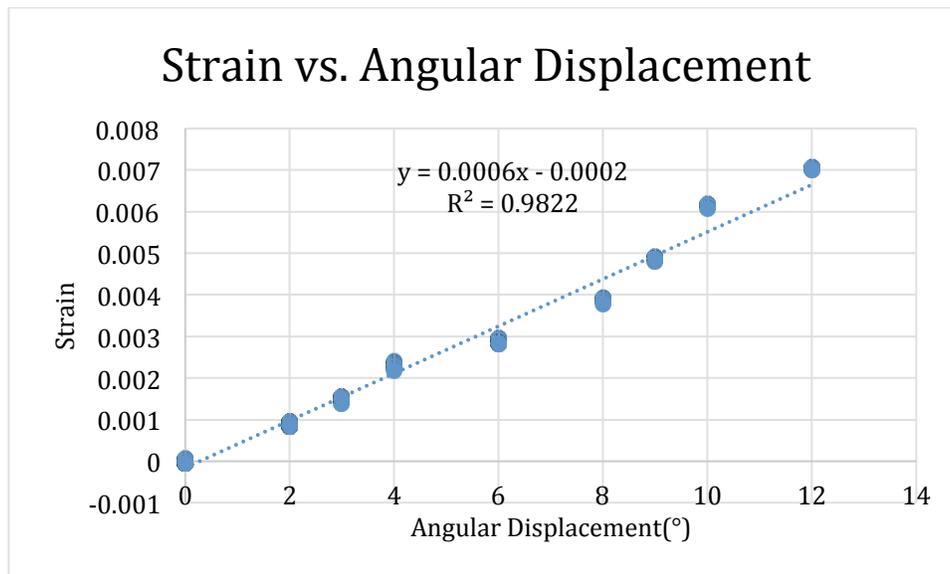


Figure 58: Strain vs. Angular Displacement

This graph depicts bending up to 12 degrees, which resulted in less than 1% strain on the beam. This percentage of strain was much less than expected since initial calculations stated that

19 degrees would result in 4% strain. The data collected was linear, which was the desired shape. Though the way his data was collected is believed to be too crude; the testing of data with an iPhone, and especially with human error of holding the iPhone, there needed to be a better way to measure the strain on the beam. A potentiometer was found to be a device that would solve this problem. The second test performed was with using a potentiometer.

Test Two

The second round of testing the mechanism was also done without the gear chain. This testing was different because a potentiometer was used to measure the displacement angle, which would give a much more reliable data than holding an iPhone. The potentiometer was attached to the bar of the mechanism by electrical tape and held with a vice grip as shown in Figure 59.

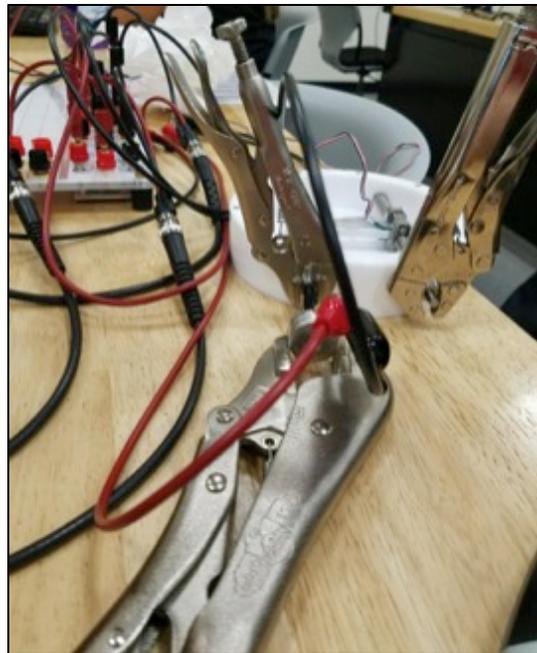


Figure 59: Testing Day Two

This testing should have given more reliable results than the first tests on the mechanism, but the data that came from these were unusable shown in Figure 60. Again vice grips were used to bend the device, so it seemed strange that the data could be so off for this testing.

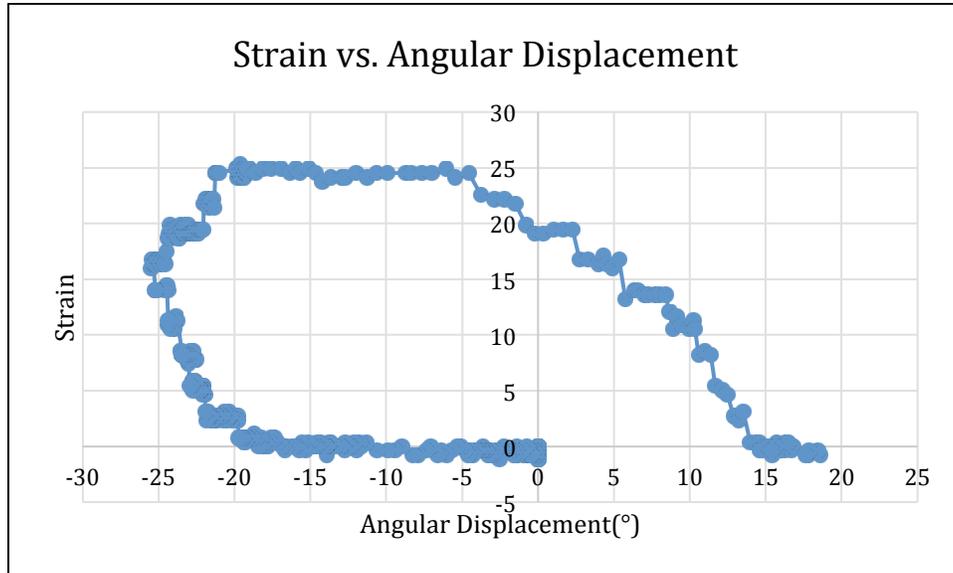


Figure 60: Testing with the potentiometer

With this other aspects of the device that could be malfunctioning, it was determined that the potentiometer was not working because of the tape that it was being attached with. When the mechanism would move, the tape would stretch but not move the potentiometer, which would not record correct angular displacement and then give the bad data received from this test. To solve this it was decided to drill a hole into the back side bars of the mechanism. This location is not needed for any other part of the experiment and would not interfere with the gear train so it was decided that this location would be the best place to attach the potentiometer so that it would get immediate strain, not delayed with the tape.

Test Three

The third trial of testing was the most successful. This trial incorporated the new method of securing the potentiometer. With the potentiometer properly fastened, there were much less variables to account for giving us more accurate data. The chamber was clamped down to the table using C-Clamps while the potentiometer was held in place using table vice. The table vice ensured that the head of the potentiometer would not rotate which would yield better results. The beam was then strained using vice grips and recorded using the same LabVIEW program. The set up for the testing is shown below.



Figure 55: Test Trail 3 Set up

The data started to get recorded when the beam was in full compression. From there the force was reversed until the beam was in full tension. Once the data was recorded and stored in an excel file, it was ready to be analyzed. The applied strain was plotted verses angular rotation. This graph is shown below

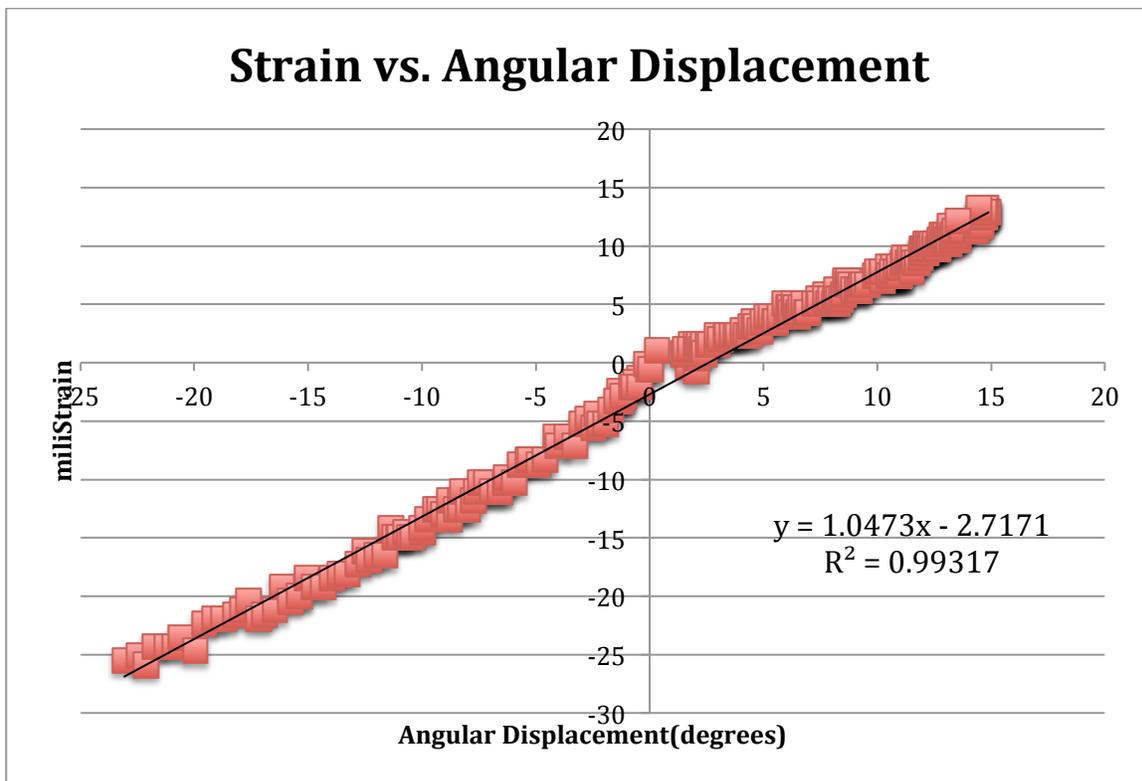


Figure 56: Strain vs Angular Displacement Graph

This graph shows a linear relationship between the applied strain and the angular rotation. This linear relation shows that the strain is uniform throughout the entire beam. Knowing this relationship the grad students that will be using this device will be able to know how many degrees the gear train must turn in order to get a desired percentage of applied strain. This graph shows millistrain for the y-axis. The axes were set so that tension was in the negative quadrant and compression was in the positive one. To convert this to percent strain the numbers shown would be divided by 1000. Once that operation is complete, instead of a millistrain of 10, it is now a percent strain of 1%.

10.4 Chamber Tests

The chamber was tested when all aspects of the mechanism were installed. When all of these parts were installed, the chamber was filled with water to make sure that it was liquid tight. Water is used first since because if the chamber weren't liquid tight and some seeps out, there would be no hazards, contrary to if acid spills out. Once the chamber is determined to be liquid tight, it is then filled with acid. The chamber was left alone for over an hour in order to test the chemical resistivity. This also tested the chemical resistivity of the mechanism and the enclosed components of the mechanism.

A second test was conducted to check if the chamber was airtight. This was done using a pressure gauge in one of the ports while everything else was plugged. If the pressure was not air tight, it would be impossible to obtain good data from this experiment.

11.0 Final Prototype Problem Solving

After completing manufacturing of the separate parts the chamber and mechanism were put together with some difficulties. Initially the O-ring that was purchased for the seal between the chamber and the lid was too small for the groove that was cut. This required a new O ring material to be bought. With the new O-ring there was a difference in depth of the cut so that parts of the O-ring had less ability to connect with the lid. This was fixed by using an adhesive to make sure that the O-ring stays in place and give a boost to the side that the groove was cut too much.

Another problem was the manufacturing of the coupler. Because of its small size and the tolerances of the CNC machines creating it, there were issues with the clearance and fitting the pieces together. These issues caused a backup of almost two weeks to put together the whole device. Luckily the pieces could be manipulated with tools, making the counter bores larger and cutting wholes larger, and put together.

Initially to hold the beam in place there would be two bars on either side of it, one would be screwed into the mechanism and the other would be held in place by a setscrew. The setscrew would use force to hold it into place, but could be removed when the user put pressure on the part. When put together and tested the setscrew could not hold the pressure needed to bend the device. It was decided that these would not work and that the best way to make sure the structure

stays together is to regular screws. We thought that we would have to create new couplers to be able to do this, but we were able to expand the drilled holes for the setscrews and fit normal screws in their place.

A major obstacle that arose during assembly was the thickness of the beam. We assumed that the beams thickness to be exactly what we ordered however this was not the case. We had ordered material that was a quarter of an inch thick but what we had received was 0.21". This was a huge difference when securing the beam since the pillars that apply the strain need to be in constant contact with the beam. To fix this issue we used aluminum shims to fill the gaps. This method secures the beam but it does add variables since they are not all the same shape or size.

As was mentioned in the Test 1 section, there was a great amount of human error that had to be accounted for. The iPhone was calibrated using the table we were operating on to ensure that zero degrees a flat position, but the iPhone was held by a person in place above one of the couplers as they were rotated, which has a lot of questionable variables. Since the gear train was not assembled at this point, the input force was also human driven creating more variables. It was impossible to have an exact strain vs rotation chart since both the applied force and the degrees of rotation were not consistent and accurate.

In the second trail a potentiometer was used in order to avoid the variables from the iPhone measuring the degrees. This seemed like a great solution since it can rotate 300 degrees and records the rotation. This also came with issues on how to properly secure it. Originally electric tape was used and the potentiometer was tightly fastened to one of the rotation rods. Even with the tape, the potentiometer was not level and would still move, causing poor displacement measurement. The tape also caused issues since it would twist when force was applied. This caused a nonlinear relationship between the strain and measured rotation, acting as a torsional spring. The tape will twist, storing spring energy until it reaches the reaction force supplied by the potentiometer at which point the spring energy is released. To solve this issue, a hole was drilled in one of the rotating titanium rods so the potentiometer could be secured.

12.0 Conclusions and Recommendations

This project required a lot of trial and error experimentation, from designs to manufacturing. A device to fit the space limit and strain the beam to the required strain needs to be machined precisely. With some of the manufacturing problems that were encountered caused the device to not perform to the requirements that we had hoped it would achieve. Throughout this project it was questionable wither this project would be successful, through the final testing it proved that it would be possible to create this device. There were many times that there was necessary research on topics that we did not know about that held up the project for weeks at a time to try and figure out how to manufacture and remanufacture the titanium and to create the beam. Having experts in these areas could help make sure that the parts would be created with the precisions and help make sure the device achieve the accuracy that is wanted for this device.

Another iteration of this device can be made in less time, and can be more precise with the information that we have collected making this device. By knowing that the mechanism

needs to be made to fit different sizes of beam, for this we tried to use set screws, but learned that the force used in this experiment is too strong for the screws. A new method of holding the beams can be researched and tested so that beams have some flexibility on their size, and no shims would need to be added. This was something that was not believed to be very important but was determined to be important to the device after testing began. The need to put in shims for the experiments would be a nuisance for each trial, and also would cause issues with the hold and slipping of the beam. A new form for this lock would be very important for the next iteration.

Something to keep in mind for the next iteration of this design is the gears that are part of the mechanism. There will be a larger amount of force on these gears so they need to be strong enough to be able to hold them. In this design plastic gears were used, but it was found that these were too weak and would break when the pressure was applied. In addition the axels that the gears were on were too weak to be able to keep the gears stiff, which is very necessary in this device. Having metal gears and smaller axels that will not move will be important in the next iteration of this device.

Also creating a way to hold the chamber and gears together is important. Adding a plate adds extra height to the device, which is not wanted with the height requirement. Creating a new system to hold the parts together that would not add any height as well as holding the system stiff is something that needs to be considered from the start. Consider making the mounting plate into the chamber itself; having the plate as the bottom surface of the chamber. This way the height would not be changed and there could be a static seal on the bottom of the chamber that would still be acid and airtight.

When the beam was strained it also would cause extreme bending of the beam, which caused the beam to move up to half an inch. This was unexpected and would cause the beam to hit the Raman spectrometer if placed underneath. Possibly a different material for the beam or a different sized beam could have fixed this problem.

Though there are many aspects of this device that need to be perfected, this device served as a method to prove a concept. Based on the data obtained from experimentation and the ensuing calculations, it was proven that the pure bending technique is a very efficient and simple method for applying the necessary strain. With more precise machining and materials expertise we believe this device can be used in ground breaking photo-electrolysis experiments. Currently there are no such devices in operation and this will be the first of its kind. Having an environmentally friendly method of harvesting hydrogen could revolutionize the renewable energy market.

APPENDIX A: Material Decision Matrix

Material	Young Modulus (Pa)	Elongation (Strain)	Water Absorption (Percent)	Durability Against UV Radiation	Durability Against 1 Mol Sulfuric Acid	Melting Point (Degrees Celsius) (Materials Processing Temp.)	Price (USD/kg)
Ionomer (zinc)	2.96e7 - 2.6e8	3.6 - 5.25	0	Fair	Satisfactory	215 - 245	3.22 - 4.21
PEBA (Shore D55)	1.45e8 - 3.49e8	4.5 - 5.48	1.14 - 1.26	Fair	Satisfactory	194 - 253	7.00 - 9.00
TPO (PP+EP(D)M, Shore D60)	6.85e8 - 7.95e8	4.51 - 7.32	0.00907 - 0.011	Poor	Satisfactory	207 - 229	3.05 - 3.52
PB (adhesive resin)	6.9e7 - 1.03e8	4.65 - 5.38	0.01 - 0.02	Poor	Satisfactory	104 - 177	2.15 - 2.37
TPO (PP+EP(D)M, Shore A90/D40)	1.78e8 - 2.36e8	4.68 - 7.55	0.019 - 0.021	Poor	Satisfactory	212 - 234	3.05 - 3.52
TPO (PP+EP(D)M, 30-32% barium sulfate)	1.16e8 - 1.84e8	4.82 - 5.19	0.00907 - 0.011	Poor	Satisfactory	204 - 224	3.15 - 3.49
TPO (PP+EP(D)M, 10-20% mineral)	1.26e9 - 1.75e9	4.82 - 5.19	0.00907 - 0.011	Poor	Satisfactory	208 - 226	3.66 - 4.09
TPO (PP+EP(D)M, Shore D50)	3.46e8 - 5.13e8	4.84 - 7.43	0.0136 - 0.0165	Poor	Satisfactory	212 - 234	3.05 - 3.52
TPV (PP+EP(D)M, Shore A85)	1.12e8 - 1.14e8	4.87 - 5.72	0.0145 - 0.0176	Poor	Satisfactory	191 - 213	3.94 - 5.76
TPV (PP+EP(D)M, Shore A85, flame retarded)	1.1e8 - 1.16e8	5.3 - 5.7	0.0145 - 0.0176	Poor	Satisfactory	188 - 198	4.60 - 6.42
TPV (PP+EP(D)M, Shore A90/D40, flame retarded)	1.22e8 - 1.28e8	5.43 - 5.87	0.0127 - 0.0154	Poor	Satisfactory	188 - 198	4.60 - 6.42
TPV (PP+EP(D)M, Shore A90/D40)	1.23e8 - 1.27e8	5.47 - 6.19	0.0127 - 0.0154	Poor	Satisfactory	199 - 220	3.94 - 5.76
SBS (Shore A90/D40)	9.75e7 - 1.03e8	5.53 - 7.73	0.0476 - 0.0525	Poor	Satisfactory	168 - 194	2.54 - 2.80
SEBS (Shore D50)	1.6e7 -	4.34 -	0.05 - 0.06	Fair	Satisfactory	192 - 218	3.97 -

	2.5e7	4.67					4.38
SEBS (Shore A90/D40)	8.68e6 - 1.08e7	5.19 - 7.03	0.05 - 0.06	Fair	Satisfactory	167 - 190	3.97- 4.38
EMA (17-25% methyl acrylate)	1.8e7 - 4.5e7	6 - 8.65	0	Fair	Satisfactory	180 - 200	2.85 - 3.14
POE/POP (Ethylene-based, Shore A65)	8.58e6 - 1.31e7	6.2 - 10.8	0.0454 - 0.0551	Fair	Satisfactory	167 - 187	2.21 - 2.52
EMA (9-14% methyl acrylate)	6.0e7 - 8.0e7	6.5 - 7.6	0	Fair	Satisfactory	180 - 200	2.85 - 3.14
POE/POP (Ethylene-based, Shore A90/D40)	7.1e7 - 8.25e7	7.01 - 9.82	0.0181 - 0.0221	Fair	Satisfactory	217 - 237	2.21 - 2.52
EVA (Shore A85, 25% vinyl acetate)	2.0e7 - 3.0e7	7.3 - 7.7	0.05 - 0.15	Fair	Satisfactory	NA	2.30 - 2.53
EBA (17-27% butyl acrylate)	2.5e7 - 4.0e7	7.3 - 9.0	0	Far	Satisfactory	190 - 210	2.85 - 3.14
PPE+PS alloy (30% graphite fiber)	7.74e9- 8.13e9	2.33-2.69	0.0364- 0.044	Fair	Satisfactory	220 - 316	11 - 13
PPS (60% glass fiber and mineral)	1.99e10- 2.09e10	2.79-3.23	0.0636- 0.077	Good	Satisfactory	269-357	8.62 - 9.51
SRP (extrusion & compression Molding)	8.30E+09	4 - 5	0.2-0.5	Fair	Satisfactory	300 -350	130 -150
LCP (30% mineral filled)	1.02e10 - 1.11e10	3.37-4.08	0.0038 - 0.0042	Good	Satisfactory	281 - 341	16.6 - 20.5
PMMA	2.7e9 - 2.9e9	0.02 - 0.07	0.02 - 0.04	Good		123 - 260	2.69 - 2.95

APPENDIX B: Beam testing procedure

EQUIPMENT LIST

- National Instruments USB-6229 DAQ
- 2310 Signal Conditioner
- Electrical Connector Plate
- Vishay Strain Gage & aluminum plate
- Shovel nose plier
- 2 feet of wire
- Blade
- Aluminum block
- Solder
- Soldering iron
- Electrical Connector Plate
- BNC "T" connector
- bnc-to-bnc Cables
- bnc-to-banana cables
- bnc-to-crocodile cables

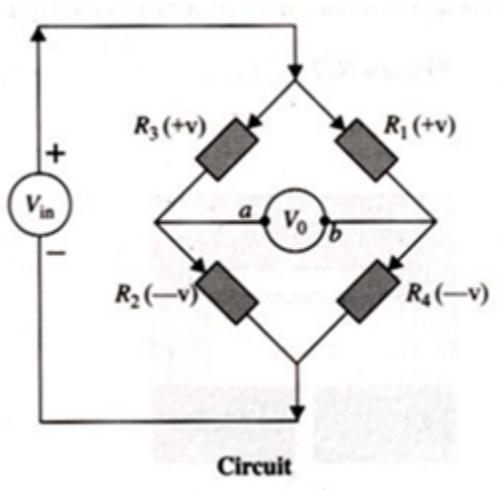
PHOTOGRAPHS



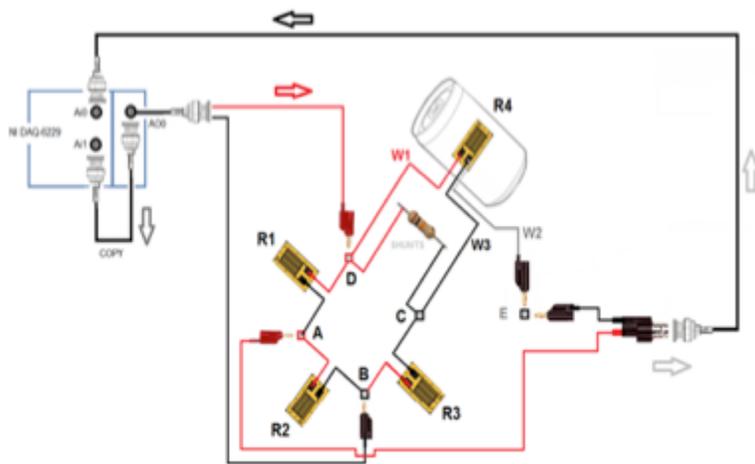
Image of National Instruments, usb 6229 bnc data acquisition box.



a SET OF bnc-TO-bnc CABLES.



Wheatstone Bridge Circuit



Wiring Diagram for Strain gage

APPENDIX C: Beam Testing Data

	Plastic	Plastic (grams)	Solvent	Copper (grams)	Mixing	Method	Speed (rpm)	Time running (s)	Curing time	Results	Conductive?
Trial 1	HIPS Pallets	1.1	Acetone	0.35	Sonific ation	Mold	-	-	48 hours +	The beam did not cure right because the mold did not have any air coming or wasn't hot enough. Not conductive.	N
Trial 2	HIPS Pallets	1.2	Acetone	0.46	Sonific ation	Mold	-	-	64 hours +	The bold was left open for air to come in, but the surface hardened and it created air bubbles inside, making the beam porous. The copper sunk to the bottom, making the bottom of the beam highly conductive but extremely brittle.	N
Trial 3	HIPS sheet/ HIPS Pallets	3	Acetone	1	Sonific ation	Spin coating	2000	40	2 hours	The plastic had a high viscosity and when it got spun it did not have a uniform coat through the entire surface of the beam.	N
Trial 4	HIPS sheet/P MMA	3	-	1	Sonific ation	Spin coating	3000	40	30 min	The speed was too fast and most of the material got thrown out of the beam, the middle did not have any coating.	N
Trial 5	HIPS sheet/P MMA	3	-	1	Sonific ation	Spin coating	2000	40	30 min	The speed was too fast and most of the material got thrown out of the beam, the middle did not have any coating.	N
Trial 6	HIPS sheet/P MMA	3	-	1	Sonific ation	Spin coating	1000	40	30 min	There was a uniform distribution of the coating throughout the beam, one layer still wasn't conductive	Y
	HIPS sheet/P MMA	0.15+sol v	Anisole	1	Sonific ation	Spin coating	2000	40	30 min		

APPENDIX D: Attachments

- LabView program
- Matlab code
- CAD programs
- CAM Programs
- Excel files from tests