ATI Specialty Alloys & Components

APPLYING ANTI-OXIDATION COATING TO THE INSIDE OF ROCKET NOZZLES

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Background information and Issues:

ATI is a company located in Oregon that makes high quality specialty metals. A current product of ATI is niobium rocket nozzles that come in 3 sizes and are often used on satellites. The manufacturing process of these nozzles involves applying a silicide coating to the nozzle to protect it from oxidation. The current method of applying the silicide coating uses a mechanical arm that dips the nozzles in a uniform manner.

The issues with the current method involve the coating "bridging" the throat of the small nozzle, shearing down, and uneven coating/undesirable thickness on the medium nozzle. The bridging has to do with the viscosity of the slurry paired with the 3 millimeter diameter hole in the throat of the small section. The large nozzle has a lot of surface area on a steep incline and gravity pulls on it as it is drying. The shearing causes an uneven coating and in order to gain the target thickness a second or third coat is often necessary. The medium nozzle does not have large issues as seen with the small and large nozzle, however, there can be some unevenness of the coating and potentially shearing. The dip method is simple and easy however it does not give the best quality coating and the coating is imperative in increasing the nozzles life.

Figure 1: Shows the three different nozzles

Proposed Solution:

For our proposed solution, we wanted to design an apparatus capable of dual-axis rotation. The two axes in question are the centerline of the nozzle, which will be referred to as the vertical axis, and an axis perpendicular to this going through the centroid of the nozzle, which will be referred to as the horizontal axis (figure 2). The idea behind the vertical axis is a continuous rotation that will induce centrifugal force, causing the slurry to move away from the nozzle centerline and thus evenly coat the interior surface. The idea behind the horizontal axis involves flipping the nozzle end-over-end, or 180o, which should counteract the shearing due to the gravitational force normally imposed upon a stationary nozzle. The constant flipping will prevent the slurry from collecting at the bottom of the nozzle, as the bottom will be in a constant state of flux. These concepts should hold true during the drying of the nozzles as well. If the apparatus is used in a drying cycle, it should prevent any of the shearing and unevenness normally present during a static dry cycle by inducing the desired behaviors detailed above.

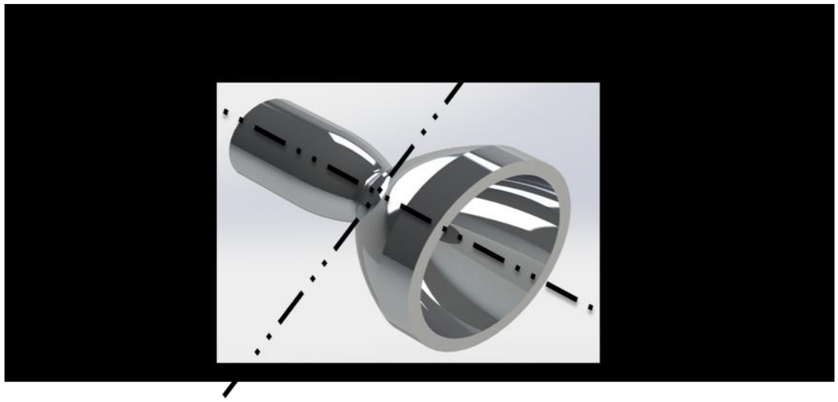


Figure 2: Shows vertices of nozzle rotation

Machining:

Medium Nozzle: The machining of the medium nozzle was the easiest and most straightforward which is why we machined it first. We ordered a large aluminum stock and cut it to the correct length. The rest of the machining was done on a lathe and with the help of our mentor we were able to utilize the CNC lathe to get a shape and contour that closely resembled the nozzle at ATI. A learning outcome of machining the medium nozzle was gaining experience on a lathe. The team had little or no experience on a lathe and machining the nozzle gave us valuable experience in taking a Solidworks model and turning it into an actual physical product via the lathe.

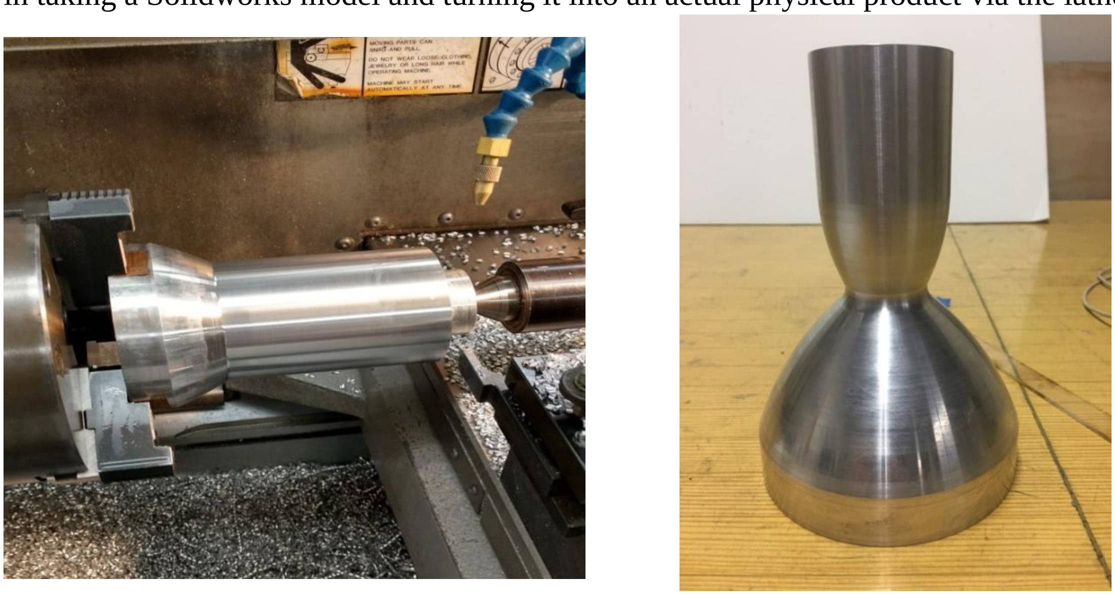


Figure 3: Shows machining the nozzle in the lathe and the final product

Small Nozzle: The small nozzle presented a unique issue with machining a nozzle with such a small throat. We special ordered a boring bar the appropriate size for our throat section, however, due to the limited reach of our new boring bar we were forced to machine the nozzle into 4 separate sections. The sections were machined with step down lips that fit together in order to press fit the 4 sections together. Learning how to adapt to the resources provided, in this case the machining tools, and still produce a nozzle that we could use for testing was a hurdle but we were able to successfully produce an accurate small nozzle.

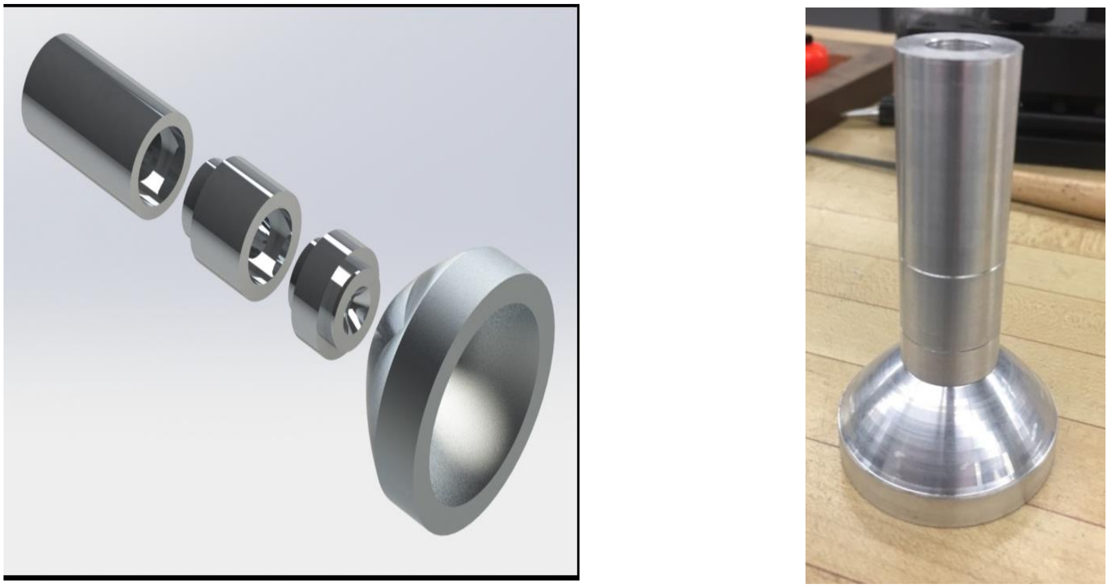


Figure 4: Shows the four different sections of the small nozzle and the final product

The apparatus: Machining the apparatus was the most time consuming process of the whole project. We started by machining the t-slot brackets drilling the appropriate holes for each section making sure to maintain concentricity between the holes for the custom shafts we machined after the t-slots. Custom brackets were made to fit over shafts and help support the weight of the apparatus so that the full weight was not on the t-slots. After receiving our bearings we machined mounting brackets for those in order to give the bearings enough space to spin freely. The 4 large aluminum plate that hold the nozzle were custom ordered. We came out of machining with a new appreciation for the small details and the importance of having an intricate model to base our machining off of. We were lucky enough to not run into any large hurdles and even though machining and putting together the apparatus took several weeks it ran fairly smooth overall thanks to help from our mentor and an accurate model to take measurements from.

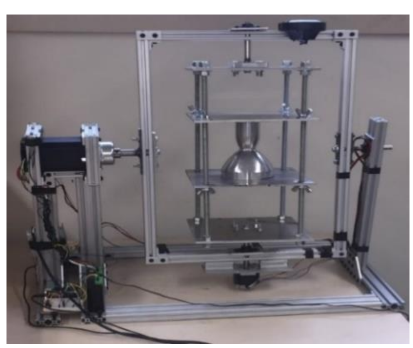


Figure 5: Shows the final product

Motors and Wiring

Small Stepper Application:

For the constant centerline rotation of the nozzle, we used a small stepper motor. Using a Microstep Driver ST-M5045, an Arduino Uno, a potentiometer, and a 12 V Power Supply with an on-off switch we created a circuit that allowed the stepper to run continuously in one direction with a variable speed. Please refer to the included circuit schematic for the specific wiring (figure

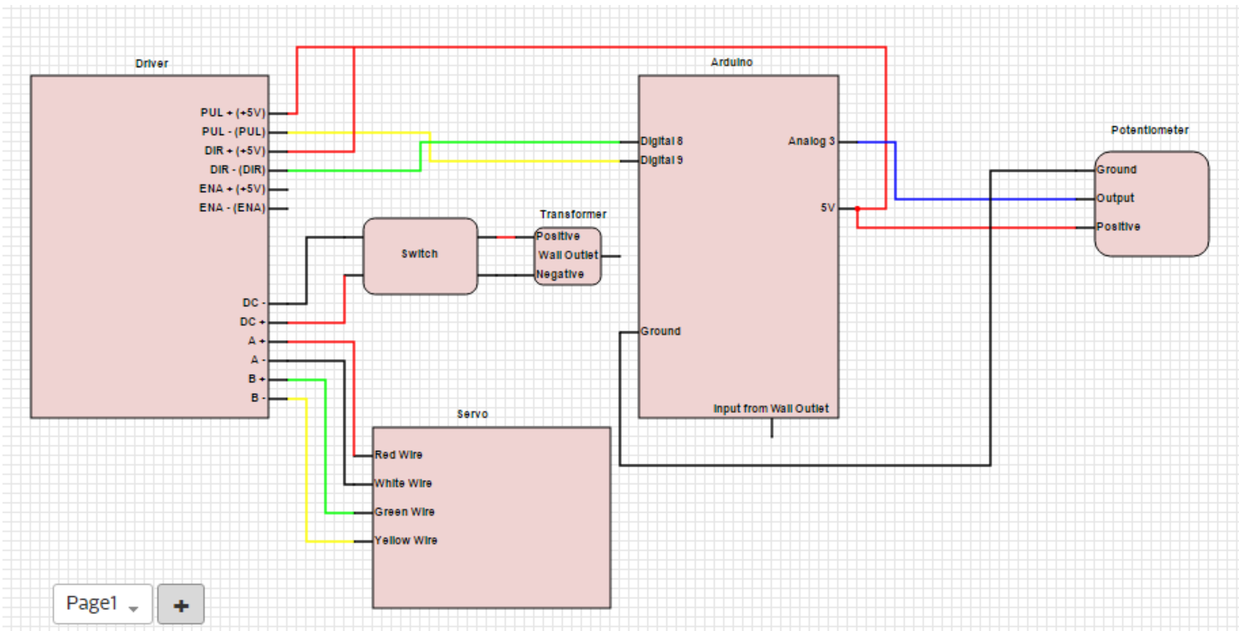


Figure 6: Shows wiring diagram of small stepper motor setup

In summary, the Arduino code maps the input from the potentiometer to a range of step values, in this case from 1023 to 3600 as shown by the highlighted numbers. Then the stepper is simply told to run at the value given from the potentiometer input continuously.

Nema-34 Application:

For the 180° horizontal rotation of the nozzle, we used a Nema-34 stepper motor. Using a Microstep Driver ST-M5045, an Arduino Uno, a potentiometer, and a 24 V Power Adaptor with an on-off switch we created a circuit that allowed the stepper to rotate 180° about the horizontal axis with a variable speed. Please refer to the included circuit schematic for the specific wiring (figure 7).

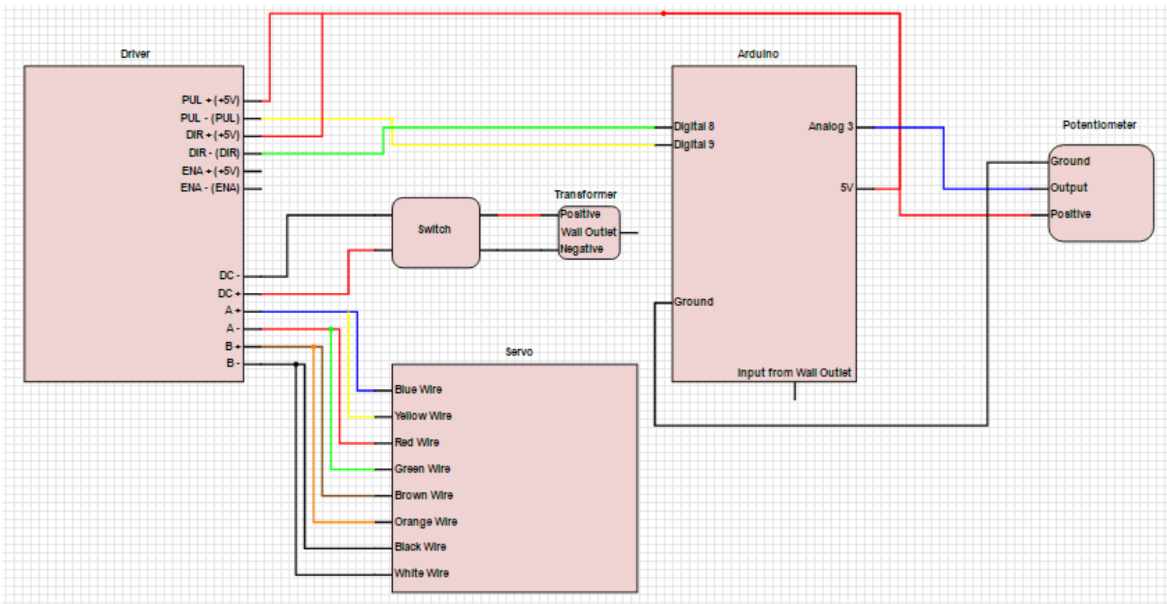


Figure 7: Shows wiring setup for Nema 34

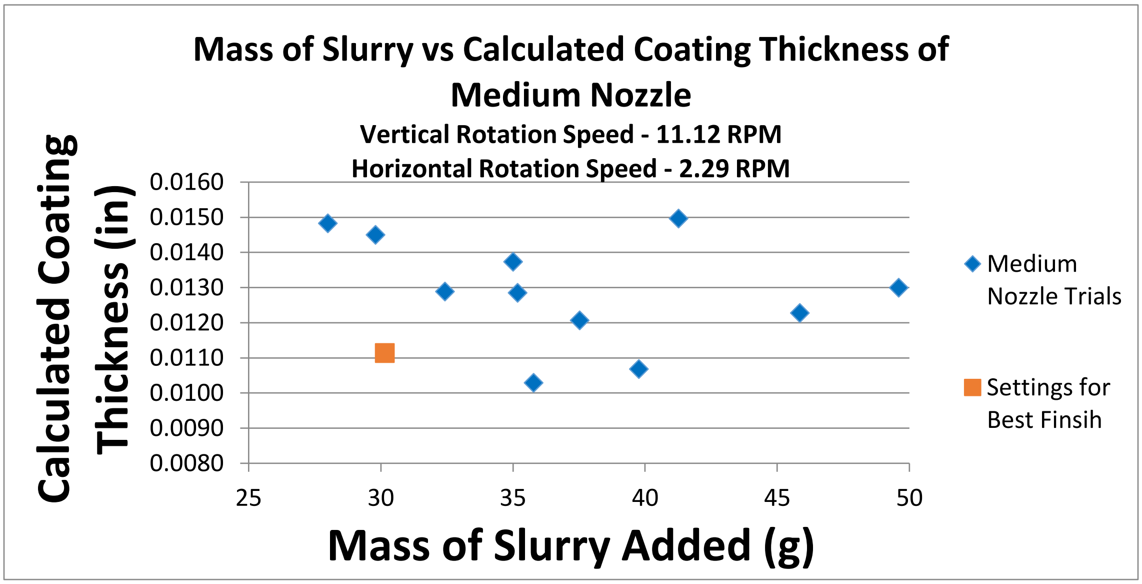
In summary, the Arduino code maps the input from the potentiometer to a range of step values, in this case from 1023 to 3600 as described in the code. Also indicated in the appendix, the code specifies the range that the stepper motor will rotate between, with 12500 corresponding to about 180°. The program runs the stepper at the speed given by the potentiometer input, and then constantly checks to see if it has gone the correct number of steps given by the red highlighted number. Once it reaches this limit, it reverses the sign of the potentiometer input and restarts the counter to continuously check if it has gone the correct number of steps given by the red highlighted number.

Medium Nozzle Best Practice Coating Technique

Preface to best practice medium nozzle coating technique: Determining the correct coating procedure was found through a variety of strategies using different sealing methods, initial slurry volumes, and different cycle times. Initial slurry weights were varied to determine the range that would successfully coat the entire inner surface of the nozzle. At the lower end, the nozzle was starved and solidified before being able to travel across entire surface as shown below.

Figure 8: Shows example of starved nozzle from too low of initial slurry weight

Below is a table of the trial results of the testing that is outlined in the above paragraph. In the beginning of testing, the hypothesis was that there would be a trend in the initial slurry weight versus the final coating thickness. As seen in the table of data and graph below, the results were very sporadic. Once lack of a trend was realized, the direction or goal of the testing turned to honing in on the correct amount of slurry that would be enough to not starve the nozzle, while not having to evacuate excess between the wet and dry coating cycles.



- Calculated Slurry Density (g/cm^3) 1.2214
- Medium Nozzle Surface Area (cm^2) 274.49
- Number of inches in cm 0.393701

Trial #	Viscosity(sec)	Initial Nozzle Weight(g)	Slurry Weight(g)	Final Nozzle Weight(g)	Calculated Coating Thickness(in)	Wet Cycle Time(min)	Dry Cycle Time(min)	Runs Present	Seal Type
1	30	876.03	5	incomplete coating	**incomplete coating**				Rubber
2	30	876.03	17	incomplete coating	**incomplete coating**				Rubber
3	30	876.03	33	884.92	**0.0104**				Rubber
4	34	876.03	41.27	888.77	**0.0150**	7	3	no	Rubber
5	33	876.03	45.85	886.48	**0.0123**	8	17	no	Rubber
6	27	876.03	49.6	887.09	**0.0130**	8	17	no	Rubber
7	29	876.03	39.77	885.12	**0.0107**	8	17	no	Rubber
8	29	876.03	35.78	884.79	**0.0103**	8	30	yes	Saran
9	23	876.03	32.43	887	**0.0129**	8	30	yes	Saran
10	23	876.03	28	888.65	**0.0148**	8	30	yes	Saran
11	29	876.03	29.8	888.37	**0.0145**	8	30	no	Rubber
12	29	876.03	35	887.72	**0.0137**	8	30	no	Rubber
13	29	876.03	35.17	886.97	**0.0128**	8	30	yes	saran
14	29	876.03	37.52	886.3	**0.0121**	8	77	yes	Rubber
15	28	876.03	30.15	885.51	**0.0111**	8	45	no	Metal

Figure 9: Shows graph and table of results. Note: saran wrap made it cleaner and avoided getting our rubber dirty and allowing dried pieces to enter the nozzle but did not allow the nozzle to dry properly even after 30 minutes.

Many tests were conducted using permanent rubber pads attached to the aluminum clamping plates, however this caused slurry to get on the rubber pads during drying cycle and resulted in uneven edge conditions and dry slurry chunks getting into future tests.

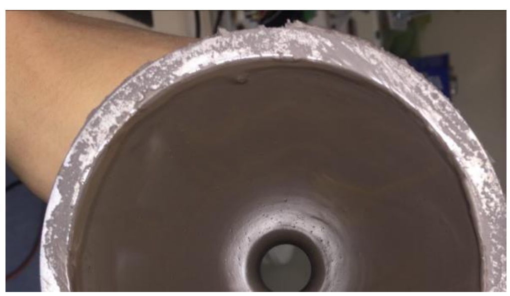


Figure 10: Shows bad edge conditions and caked on material from rubber pad

To combat this, we tried sealing the ends with saran wrap and rubber bands. During the drying cycle we would punch holes through the saran wrap to allow air to enter. Insufficient air was entering the nozzle, and resulting in massive amounts of time to obtain a dry finish.

Figure 11: Nozzle still completely wet, even after over an hour of drying cycle

Eventually we landed on this method that places the nozzle directly against the aluminum plates on both ends during drying cycle. Not only does this ensure a sharp edge condition, the aluminum plates are easily wiped clean using acetone after each use. The specific procedure and parameters are outlined in this document.

1. **Plug in apparatus using the four different wall chords, powering the two Arduinos and two drivers. Wiring diagrams and code have been provided earlier in this report for setup and troubleshooting purposes.**

2. **Shake slurry container for 1 minute by hand.**

3. **Take viscosity of slurry using ford #4 viscosity cup. During successful trial the viscosity was 28 seconds.**

Figure 12: Ford #4 viscosity cup

4. **Place nozzle on free plate and rubber pad and place on scale. Zero the scale.**

Figure 13: Shows zeroed scale

5. **Pour slurry from container into pourable measuring cup. Carefully pour desired slurry weight into the nozzle with the conical section on the bottom. Be sure during this operation to pour directly to the bottom of the nozzle and to not get any on the sides as it will likely dry before rotation can occur. <u>30.15 grams</u> is the target weight for the medium nozzle and gave us our desired coating consistency.**

Figure 14: Pour slurry into nozzle on scale until desired weight is reached

6. **Carefully transport nozzle, with plate and rubber containing the slurry into the apparatus. Place rubber pad on top of nozzle and tighten top wing nuts evenly to clamp the nozzle in place.**

Figure 15: Shows clamping down of the nozzle in apparatus

7. **Controls for vertical axis rotation located on left hand side with respect to back of large motor. Turn on vertical axis rotation using push button switch with potentiometer in the slowest position (all the way counter clockwise). Once rotation begins, speed up rotation by turning potentiometer clockwise slowly until parallel with t-slot frame (roughly 180 degrees). Speed in this location calculated to be <u>11.12 rpms</u>.**

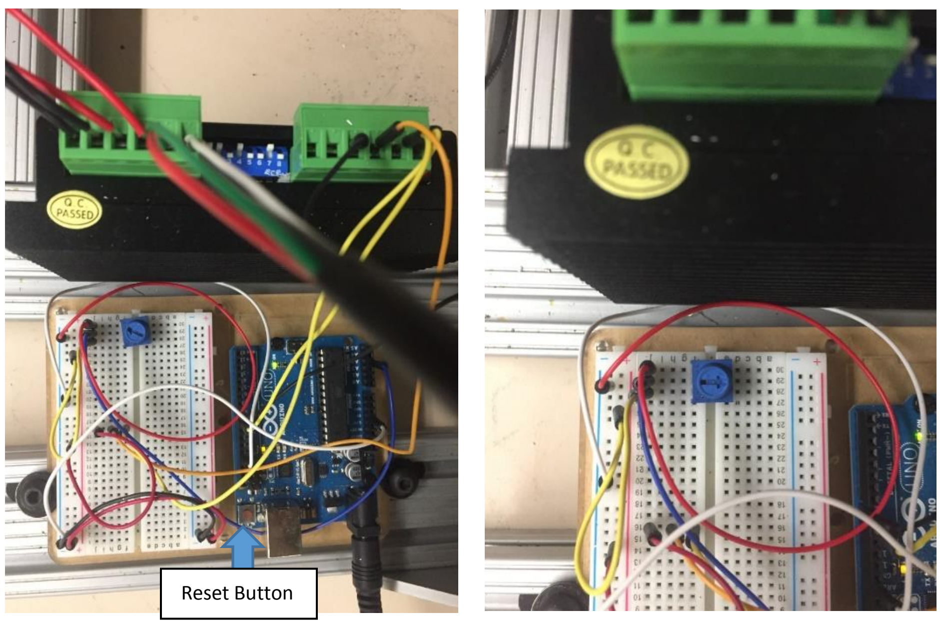


Figure 16: Potentiometer position starting the vertical rotation (left), and during cycle (right)

8. **Controls for horizontal flipping of the apparatus located on right side with respect to back of large motor. While holding the reset button on the Arduino turn on horizontal rotation using push button switch with potentiometer already in desired location (Medium nozzle – parallel with breadboard parting line). Then release the reset button on the Arduino.**

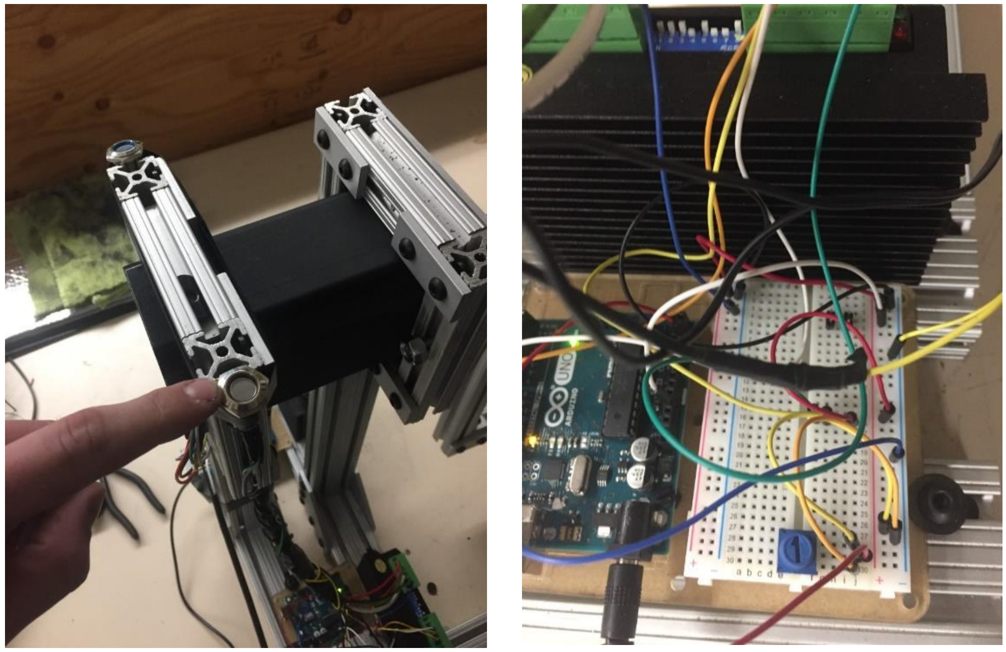


Figure 17: Shows the push button used for horizontal rotation (left) and potentiometer position for desired speed (right)

9. **Run sealed nozzle at speed parameters outlined above for <u>8 minutes</u>. Turn off both motors to stop rotation when the apparatus is in starting orientation (small motor down), and closest achievable time to 8 minutes. Loosen the wing nuts enough to be able to remove the independent plate and rubber seals without removing nozzle.**

Figure 18: Shows removing seals to transition to dry cycle

10. Re-tighten wing nuts squarely back down against the nozzle ensuring there is not gap between metal plates and nozzle due to uneven clamping. Remove Bottom plug bolt. Restart motors using procedure and speed positions as outlined in steps 6 and 7. Run unsealed drying cycle for <u>45 minutes</u>. Remove nozzle and ensure that is in fact dry. For any reason the nozzle is still wet, additional drying is required. If rotation is stopped before drying is finalized shearing will occur due to gravity.

Figure 19: Removing plug from apparatus

Successfully Coated Medium Nozzle Using Parameters outlined specifically in this "best practice" medium nozzle coating procedure.

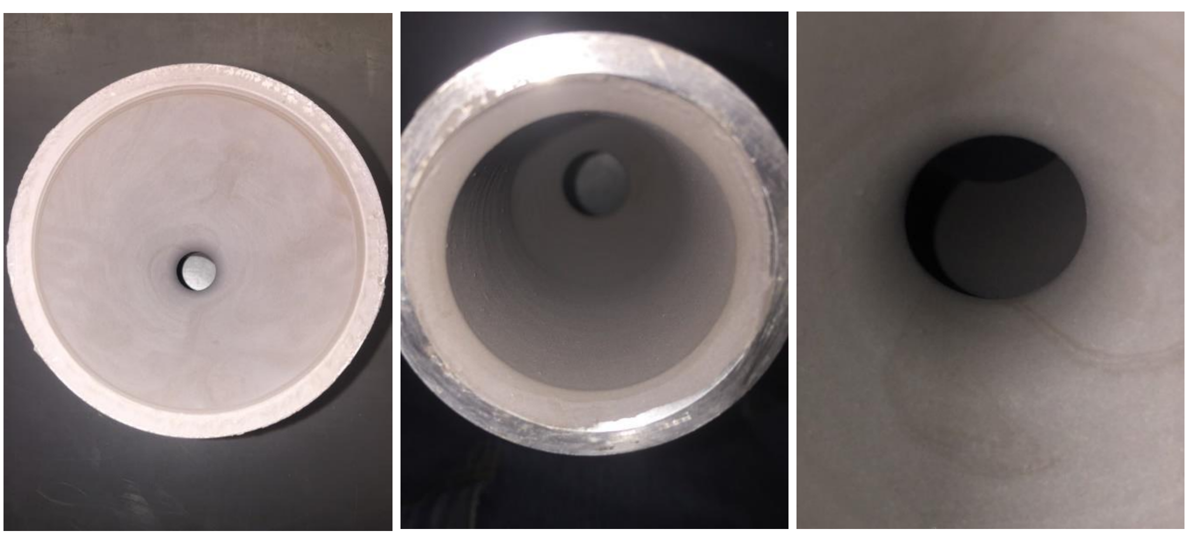


Figure 20: Successful coating of the medium nozzle

Small Nozzle Best Practice Coating Technique and Trial Structure

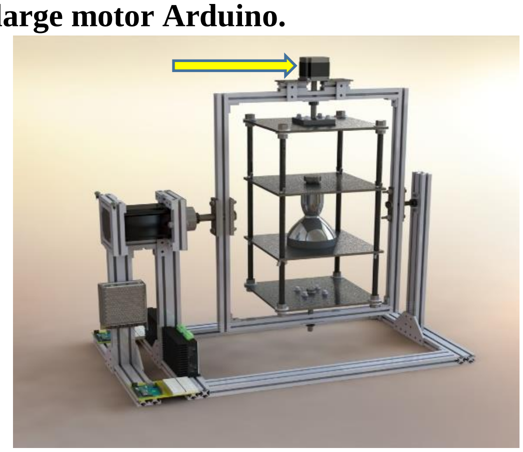
The intent of developing a coating structure for the small nozzle was to eliminate the bridging of the throat that occurs when the nozzle is simply dipped in a container as described in the background of this report. Our initial plan was to adjust all of the parameters we have at our disposal until a coating technique worked to successfully coat the interior surface while also not closing the throat.

Initially, the starting slurry weight was varied to see if it had any impact on the bridging at the throat. After several tests with different weights, we soon realized that mostly because of its size, no matter how much or how little slurry we put into the nozzle, bridging always occurs.

With this new found knowledge, we decided to change the filling method used with regard to the small nozzle, instead of having a wet cycle and then a dry cycle, only a drying cycle will be utilized with the small nozzle. As outlined in the specific procedure below, we began to hold the nozzle above the container and slowly pour the slurry through the nozzle, coating the entire inner surface, letting the excess run back into the container. While we were filling the nozzle this way, we made a discovery that would eventually lead us down a path to successfully coating the nozzle.

We discovered that the bridging only occurs when the conical section of the nozzle is below the thrust chamber. When the still wet slurry runs from the thrust chamber down into the throat, it plugs. However when you turn the nozzle 180 degrees back around, with the conical section facing upwards, it has sufficient air to funnel the slurry back through the throat and unplug. Because of this discovery we decided to limit the horizontal rotation to around 0-100 degrees rather than the full 180 degree rotation that resulted in successful coating on the medium nozzle. This technique, at the same speeds as the medium nozzles, produced many successful small nozzle coatings without throat closure. The detailed steps of the process are outlined in the best practice coating procedure below.

1. **Plug in apparatus using the four different wall chords, powering the two Arduinos and two drivers. Wiring diagrams and code have been provided earlier in this report for setup and troubleshooting purposes. Orient apparatus so small motor is at top of rotation (pointing up) and make sure correct code is loaded into large motor Arduino.**



Arrow shows initial position of small motor

2. **Shake slurry container for 1 minute by hand.**

3. **Take viscosity of slurry using ford #4 viscosity cup. During successful trial the viscosity was 29 seconds.**

4. **Hold small nozzle over can of slurry with thrust chamber up. Pour slurry into thrust chamber until completely full. Turnover and repeat for conical section being careful not to overfill allowing slurry to run down the outside of the nozzle.**

Figure 21: Shows the fill method for the small nozzle

5. **Let excess slurry run out of nozzle and into can to limit excess runoff while rotating in apparatus. Insert nozzle into apparatus conical section up.**

Figure 22: Shows nozzle being inserted into apparatus

6. **Controls for vertical axis rotation located on left hand side with respect to back of large motor. Turn on vertical axis rotation using push button switch with potentiometer in the slowest position (all the way counter clockwise). Once rotation begins, speed up rotation by turning potentiometer clockwise slowly until parallel with t-slot frame (roughly 180 degrees). Speed in this location calculated to be <u>11.12 rpms</u>.**

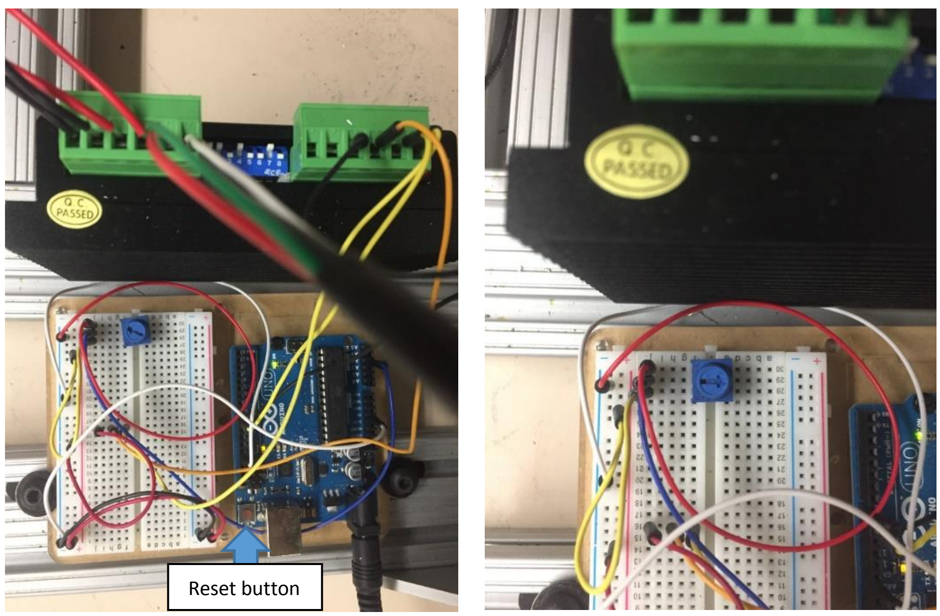


Figure 23: Potentiometer position starting the vertical rotation (left), and during cycle (right)

7. **Controls for horizontal flipping of the apparatus located on right side with respect to back of large motor. While holding the reset button on the Arduino turn on horizontal rotation using push button switch with potentiometer already in desired location (Small nozzle – parallel with breadboard parting line). and then release the reset button on the Arduino**

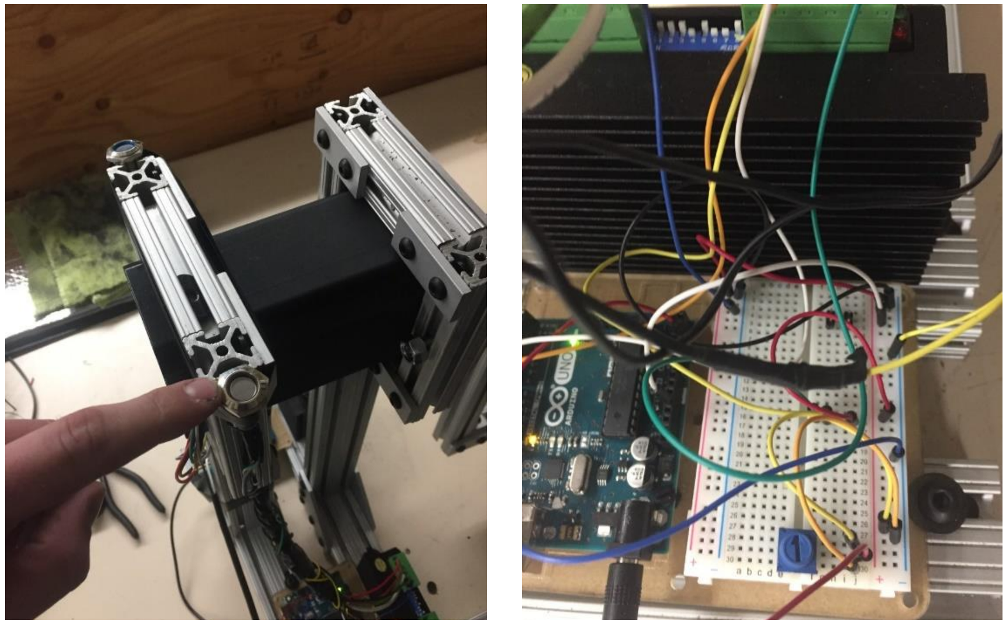


Figure 24: Shows the push button used for horizontal rotation (left) and potentiometer position for desired speed (right)

8. **Run apparatus for approximately 30 minutes ensuring proper rotation of 0 to 100 degrees. Once apparatus is back in starting position (small motor up) turn off both motors by pressing the push buttons.**

9. **Remove and inspect nozzle. These are the parameters that gave us a successful coating**

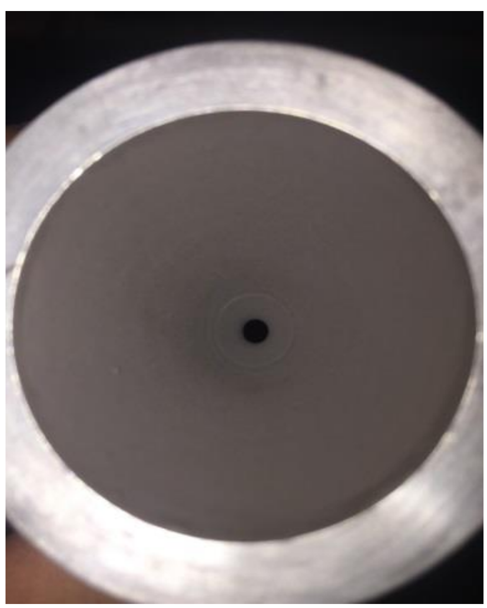


Figure 25: Shows final coating for small nozzle

<u>Overview of Outcomes</u>

Medium Nozzle:

As outlined in the trial structure section of this report, we were able to successfully establish coating parameters for the medium sized nozzle. The specific numerical parameters are listed in the table below. This is not to say that these values cannot be optimized further through additional testing. If different coating thickness is desired, these parameters will need to be altered to achieve it.

Successful Medium Nozzle Coating Parameters	**Values**
Slurry Viscosity	***28 (sec)***
Initial Slurry Weight	***30.15 (g)***
Wet Sealed Cycle Time	***8 (min)***
Dry Cycle Time	***45 (min)***
Vertical Axis Rotation Speed	***11.12 (rpm)***
Horizontal Axis Rotation Speed	***2.28 (rpm)***
Horizontal Axis Rotation Angle Limits	***0-180 (deg)***
Calculated Coating Thickness	***.011"***

Figure 26: Medium nozzle before coating

Figure 27: Medium nozzle after coating

Small Nozzle:

The outcome with respect to coating the small nozzle was determining correct angular rotation limits due to a discovery that bridging of the throat only occurring at a certain orientation. When the conical section is below the thrust chamber, bridging occurs. Thus rotational limits were limited to from 180 degrees to around 100 degrees. Also, because of the plug hole sizes versus the thrust chamber size of the small nozzle, the loading orientation of the small nozzle is 180 degrees from that of the medium nozzle.

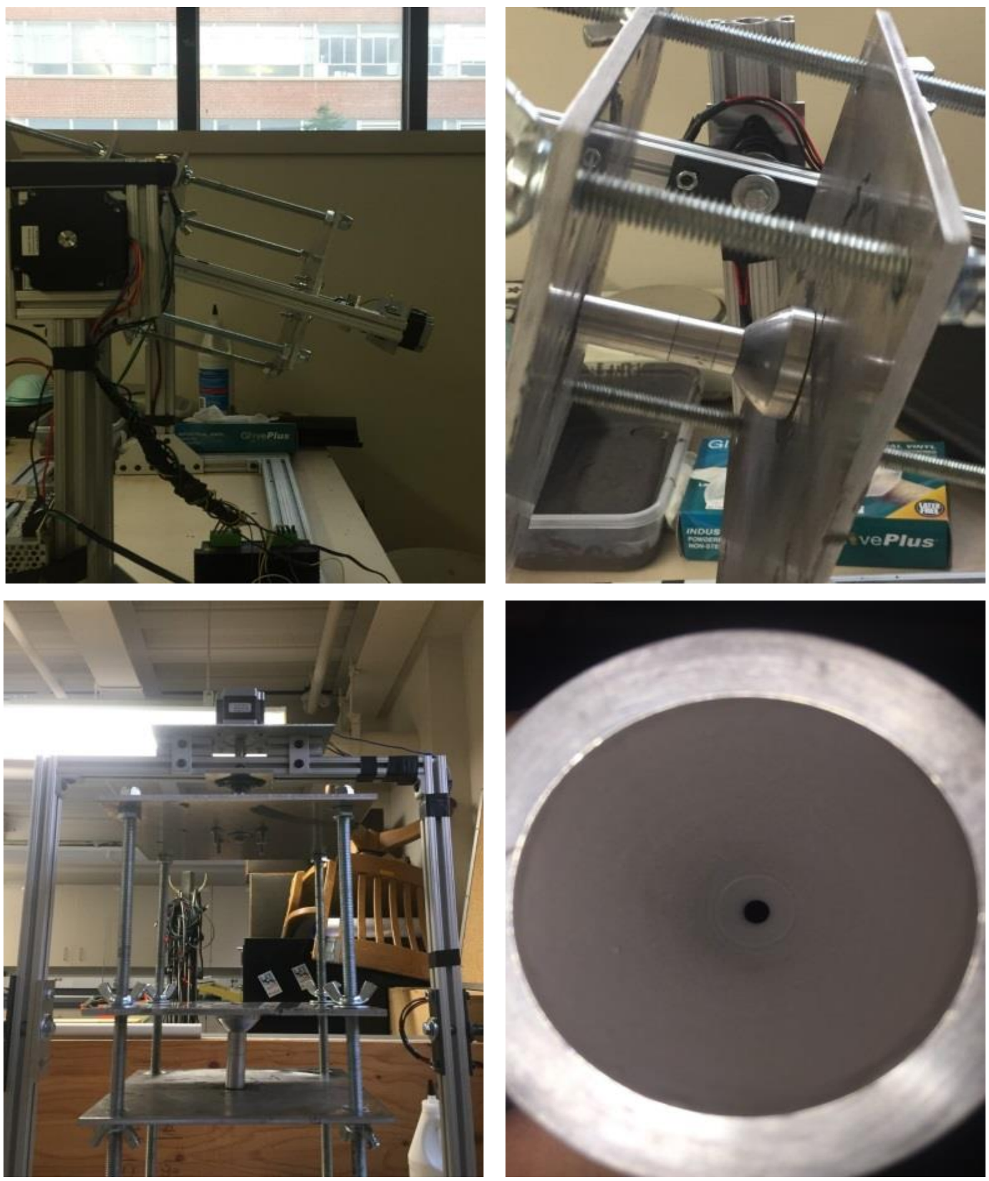


Figure 28: These four pictures show the adjusted angle of horizontal rotation, correct loading orientation and successfully coated nozzle

Future research:

Due to the nature of our testing we were unable to machine the large nozzle and perform specific tests coating the inside of the large nozzle and look at shearing. Even though we did not get to specifically test the large nozzle we believe that the results we gathered from the medium nozzle can be transferred to the large nozzle and theoretically with our rotation the shearing would be eliminated. All that said, actual testing could be done to prove our hypothesis.

Refining the parameters for each nozzle could be the next step in optimizing our apparatus. We concluded with some good baseline parameters but further research could refine these. For example the initial amount of slurry could be refined to a tenth of a gram and the optimal wet and dry cycle times could be found to have a more finite tolerance rather than a tolerance of plus or minus a minute.

The other aspect that could be looked at is the ease of use of the apparatus itself. We did some preliminary research into a quick clip system but never actually got to integrate it into our design which would make the adjustment for different nozzles much quicker. We also looked into different potentiometers but could not find a potentiometer that gave a good readout and worked well with the motors. Finding a potentiometer paired with a digital readout of speed could refine the speed parameters for each nozzle and increase the repeatability of the apparatus and its function.

Appendix A: Both codes will be attached separately, however it can also be directly copied from this document

Vertical axis Arduino code: Same rotational speed was used for both the small and medium nozzles.

```
/*
  microstep driver ST-M5045
  Pul+ goes to +5V
  Pul- goes to Arduino Pin 9
  Dir+ goes to +5V
  Dir- goes to to Arduino Pin 8
  Enable+ to nothing
  Enable- to nothing
*/

int sensorPin = A3; //This assigns the variable 'sensorPin' to A3. sensorPin is the output from the potentiometer
int sensorValue = 0; //This initializes the 'sensorValue' as 0. sensorValue is the input into the servo after conversion from degrees to steps.

void setup() {
  pinMode(8, OUTPUT); //direction pin
  pinMode(9, OUTPUT); //step pin
  digitalWrite(8, LOW);
  digitalWrite(9, LOW);
}

void loop() {
  sensorValue = analogRead(sensorPin); //This reads the angle from the potentiometer and stores it in 'sensorValue'
```

sensorValue = map(sensorValue, 0, 1023, 3600, 1); //This maps 'sensorValue' from degrees to steps. The values '1023' and '3600' set the lower and upper limits for the step speed, respectively. If you want to change the speed limits you would change these two values.

```
  //The following 4 lines set the stepper to step at the speed defined with 'sensorValue'
  digitalWrite(9, HIGH);
  delayMicroseconds(sensorValue);
  digitalWrite(9, LOW);
  delayMicroseconds(sensorValue);
}
```

Horizontal axis Arduino code: Instructions included for what values to alter from small to medium nozzles.

```
/*
  microstep driver ST-M5045
  Pul+ goes to +5V
  Pul- goes to Arduino Pin 9
  Dir+ goes to +5V
  Dir- goes to to Arduino Pin 8
  Enable+ to nothing
  Enable- to nothing
*/
```

int sensorPin = A3; //This assigns the variable 'sensorPin' to A3. sensorPin is the output from the potentiometer

int sensorValue = 0; //This initializes the 'sensorValue' as 0. sensorValue is the input into the servo after conversion from degrees to steps.

int Distance = 0; //This initializes the 'Distance' as 0. Distance is used to count how many steps the stepper has taken.

```
void setup() {
```

```
  pinMode(8, OUTPUT); //direction pin
  pinMode(9, OUTPUT); //step pin
  digitalWrite(8, LOW);
  digitalWrite(9, LOW);
}

void loop() {
  sensorValue = analogRead(sensorPin); //This reads the angle from the potentiometer and stores it in 'sensorValue'
  sensorValue = map(sensorValue, 0, 1023, 3600, 1); //This maps 'sensorValue' from degrees to steps.
  //The values '1023' and '3600' set the
  //lower and upper limits for the step speed, respectively.
  //If you want to change the speed limits you would change these two values.

  //The following 4 lines set the stepper to step at the speed defined with 'sensorValue'
  digitalWrite(9, HIGH);
  delayMicroseconds(sensorValue);
  digitalWrite(9, LOW);
  delayMicroseconds(sensorValue);

  Distance = Distance + 1; //Every time the program passes this point, it adds 1 to the 'Distance' step counter

  //The following 'if' loop checks the number of steps that have passed during operation
  if (Distance == 12500) //This value of 12500 signifies the end of the counting loop,
    //and also the end of the motion of the apparatus. It correlates to roughly 180 degrees.
   //The value used for roughly a 100 degree limit (used for the small motor) is 8000.
   //Changing this value will alter the amount of steps the stepper will take during the cycle,
```

```
  //and thus the degrees of rotation for the apparatus during one oscillation.
  //At this point the program checks to see if we have reached the step limit
 {
  if (digitalRead(8) == LOW) //If we have reached the step limit, this 'if' and 'else' loop will flip the direction of rotation
  { //The 'if' loop changes the direction from negative to positive
    digitalWrite(8, HIGH);
  }
  else //The 'else' loop changes the direction from positive to negative
  {
    digitalWrite(8, LOW);
  }
  Distance = 0; //This resets the step counter to 0 after every completed count
  delay(500); //This makes the system wait 0.5 seconds before beginning the next count cycle.
              //For the small nozzle, we used a 2 second delay at each limit, thus a value of
              //2000 will need to entered for the small nozzle
 }
}
```

Appendix B: Part Numbers & Vendors

Part Name	Purpose	Part Number	Vendor	Link
Large stepper	horizontal rotation	KL34H2160-62-8A	Automation Technology Inc.	https://www.amazon.com/Single-Shaft-Stepp
Small stepper	vertical rotation	13656	Sparkfun	https://www.sparkfun.com/products/13656
Small bearing	support and smooth operation	1ZGN3	Grainger	https://www.grainger.com/product/DAYTON
Large bearing	support and smooth operation	1A402	Grainger	https://www.grainger.com/product/DAYTON
12v power supply	power small stepper	323337	Jameco Electronics	http://www.jameco.com/webapp/wcs/stores/
24v power supply	power large stepper	174879	Jameco Electronics	http://www.jameco.com/webapp/wcs/stores/
arduino	run code to motors	50	Adafruit Industries	https://www.adafruit.com/product/50
driver	communicate from arduino to motor	10201	Sainsmart	https://www.amazon.com/SainSmart-Micro-
on off switch	turn motors on/off	917	Adafruit Industries	https://www.adafruit.com/product/917

Actual excel file will be attached electronically along with this document, so the links may directly clicked on from there.

Appendix C: Pertinent Photographs

Figure 29: This picture shows poor edge condition from using rubber seals

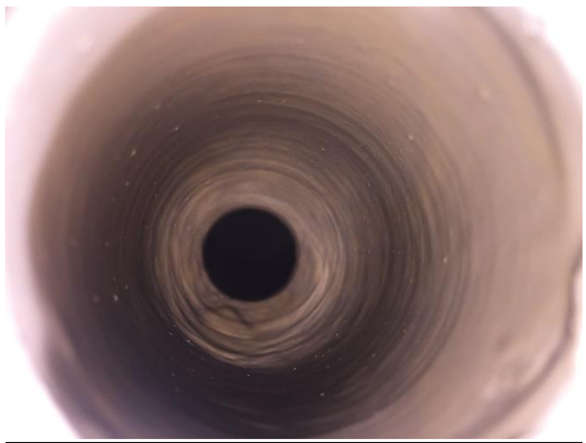


Figure30: This picture shows shearing and sludge in throat from removing before dry and possibly too much initial slurry volume

Figure 31: Nozzle is starved from not enough initial slurry volume

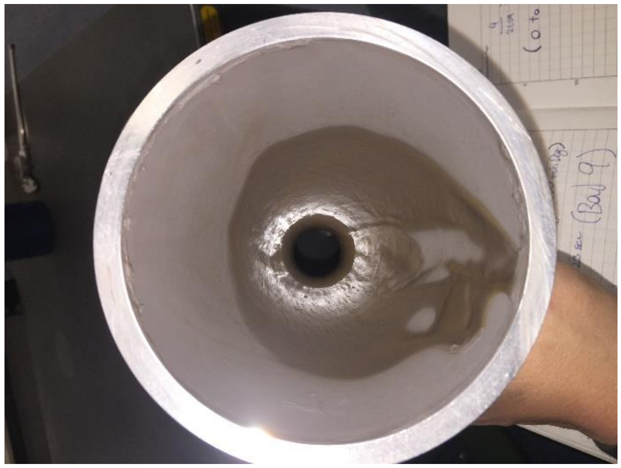


Figure 32: Shearing occurred during this test from choosing much too short dry cycle time

Figure 33: Combination of shearing and starvation shown here

Figure 34: We believed the striations seen here are a result of the slurry getting water in it, causing a rust of sort