Diesel Particulate Generation

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Abstract

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The goal of this project is to design a diesel particulate generator to test aerogels as diesel particulate filters. Diesel particulate generators are machines that generate diesel particulate matter, or diesel soot. Diesel particulate filters have been recently put into the market to help many car companies meet the ever-changing diesel emission regulations set by the EPA and other international agencies. The main goal of the filter is to block soot from exiting to the environment. Once the filter is full, the soot is oxidized and released. The problem with these filters is that they are large, costly, detrimental to fuel efficiency, and can sometimes melt at high temperatures. Aerogels are a potential solution to this problem as they can hold just as much if not more soot as current filter designs, while having a smaller surface area and being able to withstand high temperatures. These advantageous characteristics would likely combine to allow for filter designs that are less detrimental to fuel efficiency. Past research by Union College Alumnus, Jacob Cetnar, has proven that aerogels can trap diesel particulate matter and that further research needs to be conducted. While there are commercial ways to test diesel particulate filters, they are all built for full scale industrial testing, something that is not desired by the Union College Aerogel lab. Therefore, some sort of method for loading aerogel samples with soot in a Union College laboratory setting needs to be developed. The system described in the following report, consists of an air gun blowing 3 cfm of air and diesel soot simulant through 26 inches of 316L Stainless Steel tubing with a diameter of 1”, into a test cell, a 1” diameter, 3.52-inch-long tube, that will house the aerogel. The powder is introduced through a funnel and auger system that is powered by a 3V DC motor. All tubing is part of a quick clamp system where quick clamps are used to connect the flanges of the tubes together.

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1. Introduction/Background

1.1 Diesel Engines

Diesel engines are used in a wide variety of applications and have intriguing qualities. Diesel engines are superior on gas mileage, with some engines improving gas mileage by more than 20% when compared to their gas counterparts [1]. Additionally, diesel engines generally produced more torque when compared with gasoline engines, which translates into increased towing capabilities of large cars and trucks [1]. Diesel engines are also inherently more reliable than gasoline engines [2]. Since there are no sparks involved in a diesel engine, there is less required maintenance and therefore a longer service life for the engine overall. Furthermore, newer diesel engines are becoming increasingly quieter than past generations which are making them more comparable to gasoline engines [2]. However, just like with gasoline engines, there are tradeoffs. One of the biggest is the by-products of diesel combustion, or diesel particulate matter (DPM).

1.2 Diesel Particulate Matter

Diesel particulate matter is a part of diesel exhaust that contains soot particles. These soot particles are carbon based and additionally contain a combination of metallic abrasives, ash, sulfates and silicates [3]. Within these subcategories, DPM is known to contain over 40 different toxic components, including carcinogens such as benzene, arsenic and formaldehyde [4].

Beginning in the mid 1980's in the United States, lawmakers and Environmental Protection Agency (EPA) officials began recognizing the problem with diesel emissions. Lee Thomas, then administrator of the EPA, set out to put standards in place to regulate diesel emissions in cars and trucks [5]. The first wave of standards was to try and reduce the amount of emissions rather than filter them. This came to fruition when the EPA was successful in reducing the level of sulfur in diesel fuel to 500 parts per million (ppm) from 3,000 ppm in 1992, dropping diesel emissions [5]. Additionally, in the early 1990's amendments were made to the Clean Air Act that set goals of reducing emissions in phases. Originally scheduled to start in 2004, DPM reduction technology began being research in 2002. At the same time, DPM levels were set by the EPA at 0.1 gram per horsepower-hour (g/hp-hr) [5]. By 2007, car companies were unable to keep up with the exhaust regulations set by the EPA without the use of some sort of filter. Most cars and trucks, post 2007, now come with a diesel particulate filter (DPF) to further reduce the DPM output as a result of EPA regulations [5].

1.2 Diesel Particulate Filters

One of the main types of diesel particulate emission technology is the diesel particulate filter. A diesel particulate filter plays a similar role to the gasoline

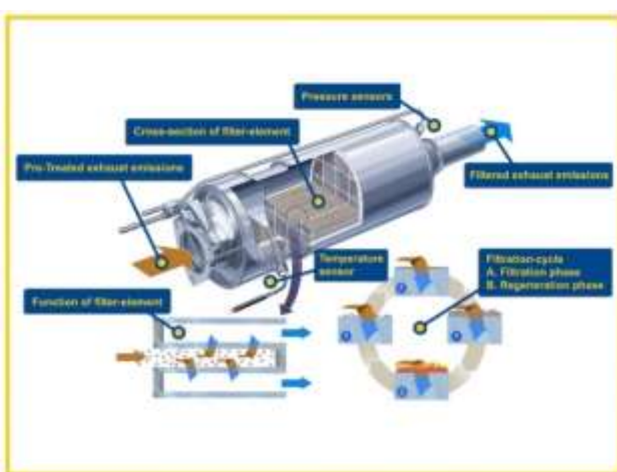

Figure 1 Labeled diagram of a diesel particulate filter system [20].

engines catalytic converter counterpart; however, it works in a different way. Figure 1 is a section view of a typical diesel particulate filter. The most common material for these filters is cordierite. Cordierite is a ceramic material that acts as a good filter with a low pressure drop, and low thermal expansion qualities [6]. A draw back to this material, however, is that cordierite has a low melting point (about 1200^{o}C) which has led to some substrates melting during the regeneration process [7]. Cordierite DPF's are a type of wall flow filter. Referring to Figure 1, diesel exhaust is pretreated and then enters the filter chamber. Once in the chamber, the exhaust encounters the cordierite wall flow filter. Figure 2 shows a close of a typical wall flow filter. In a DPF system the exhaust travels into the channels of the filter (blue arrows) [6]. The idea is that the diesel particulate matter will stick to the walls of the filter while gases escape through the walls (gray arrows) [6]. After the filter has captured the soot particles, the DPF will be covered in the black soot. Ideally, the only things that escape out the exhaust are H_2O and CO_2 [6]. However, other gases including carbon monoxide and nitrous

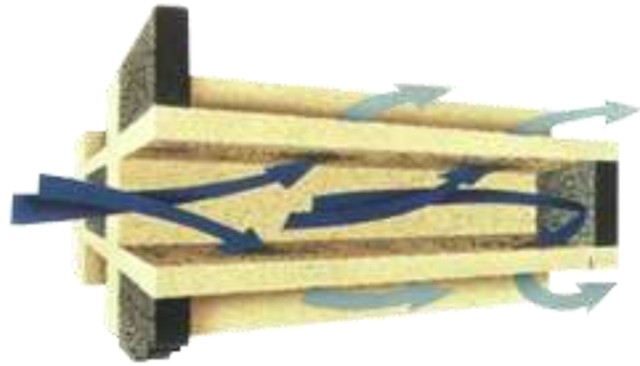

Figure 2 A basic wall flow filter [7].

Figure 3 Diesel Particulate Filter covered in soot, in need of regeneration [10].

oxides. After this process the filter will look something like Figure 3 a DPF covered in soot, needing to be cleaned. Pressure sensors at the back of the filter alert the cars computer system when the filter is becoming clogged. When the sensors are alerted, the computer automatically heats up the filter to temperatures around 600^{o}C [7]. This occurs by fuel being injected and then ignited in the filter chamber [7]. Although effective at achieving high temperatures, the use of extra fuel significantly reduces fuel efficiency. At this temperature, the soot is oxidized and then passes through the filter and out the exhaust leaving a clean and operational filter. This process is called active regeneration [7] and needs to occur relatively frequently to ensure the efficiency of the DPF. In addition to the fuel efficiency penalty, regeneration must take place when the car is travelling over 35 mph or when the engine is at the best running temperature, therefore when the car is in low speed conditions regeneration cannot occur. This can lead to blockage problems which can turn into costly repairs and replacements for the owner [7].

Although the diesel particulate filters that are available now are effective, some being 99% effective by particle count [7], they are limited. Most of these limitations are associated with everyday vehicles and regeneration. As with most problems, test methods and solutions follow.

1.3 Diesel Particulate Generation and Filter Test Methods

There are currently two fundamentally different methods commercially available to companies looking to test

Figure 4 A typical engine dynamometer [22].

diesel particulate filters, both of which are full scale testing and extremely expensive. The first of these methods is the use of dynamometer and an actual diesel engine. This is an alluring option for many car manufacturers and DPF testing facilities because it will provide the user with a realistic simulation of what will happen to the DPF in road conditions. However, these systems are large and expensive. Some systems, used by large companies such as GM, can cost upwards of millions of dollars [8]. Additionally, these systems generally take a lot of staff and immense training time to run a successful test. Again, for large companies who are able to do their own testing, this life like test is worth the investment, however, which is not the case for every test application.

While the engine dynamometer is one of the most utilized methods, a company based in England, Cambustion, has developed their own method. Cambustion's *DPG* is a full scale diesel particulate filter testing system that offers very accurate results per Cambustion. When compared to an engine dynamometer, this system is much cheaper and much more accurate [9]. Additionally, the *DPG* is extremely easy to use and requires little to no human interaction as the computer will handle most of the testing, saving money and resources while running tests [9]. This system can mimic high temperature road

Figure 5 Cambustions DPF Testing System, the DPG [9].

conditions as well as cold flow tests for other applications [9]. Due to the price of this system, many companies will send their DPF's over to get them tested rather than purchasing their own dynamometer.

While both of these systems have proven to be useful for large companies, they are not quite applicable for a small research institution such as Union College. Testing for potential diesel particulate filters is a research interest for members of the Union College Aerogel Lab (discussed more in Section Aerogel's as Diesel Particulate Filters) and for those tests to happen, they need to be scaled down to an appropriate size. This means that it is not practical for Union to either invest in an engine dynamometer nor send samples to Cambustion, therefore, some sort of method needs to be developed that is suitable for Union's Aerogel lab, but first, why aerogels?

1.4 Aerogel's as Diesel Particulate Filters

Union College Alumnus, Jacob Cetnar '17, researched aerogels as potential diesel particulate filters for his senior project in 2016. Jacob started out by testing silica aerogels which can be up to 99% air by volume. Additionally, they have a porosity of about 75% [10] resulting in surface areas ranging from 500-1200 m^2/g [11]. This range of surface area makes silica aerogels an intriguing candidate as a diesel particulate filter because larger surface areas correlate to better

Figure 6 Soot covered aerogel [12].

filter efficiencies. If silica aerogels offer higher surface areas then theoretically they can hold more soot particles which would reduce the amount of times the filter would have to be regenerated, saving extra fuel [12].

With these intriguing qualities, Jacob designed a test section which would allow soot from the exhaust of a diesel truck (without a previously installed DPF) to flow through silica aerogel granules. Figure 6 is the aerogel filter after the soot loading from the truck. Clearly, this demonstrates that the silica aerogel captures a significant amount of soot. This filter was then heated up to approximately 600^{o}C to simulate regeneration [12]. Again, the aerogel behaved as a normal filter and all soot was burned off. Furthermore, mass measurements of the aerogel filter were taken before and after the regeneration process and it was found that 4.34 grams of soot was burned off [12]. When Jacob scaled his model up to normal DPF sizes, it was found that a full size silica aerogel filter could theoretically hold between 70-90 grams of soot [12], a higher range than the typical DPF of 30-80 grams [13]. These results are promising to say the least; however, further testing on aerogels definitely needs to be done. Since the methods mentioned in Section 1.3 are not suitable for aerogel testing, a new method needs to be developed.

Figure 7 Aerogel that has been regenerated [12].

1.5 Project Goal: Design a Diesel Particulate Generator

As evident by Jacob's senior project last year, aerogel could not only be an alternative to cordierite DPF's but a better one. However, more extensive testing on aerogels needs to be done and in order to do this a method needs to be developed for the Aerogel lab at Union. Theoretically, the Aerogel lab could create a full-size silica aerogel filter and send it off to Cambustion for DPF testing; however, the resources are not available at Union to make an aerogel filter that large. Additionally, for that same reason, it makes no sense to invest in a full-size engine dynamometer because there would be no way to test a scaled down filter in it. Therefore, some sort of scaled down testing mechanism will need to be developed for further testing.

The goal for this project is to research previously used lab-scaled test methods and design one for the use of the Union College Aerogel lab. Furthermore, the designed diesel particulate generator should be able to generate soot, or a soot substitute, in a constant manner. The design should also alleviate any problems of soot clogging and should ensure that the maximum amount of soot is reaching the aerogel filter. Additionally, each test done on this DPG should be repeatable. The following report will outline the design process and final design of the diesel particulate generator. It will also discuss preliminary testing results as well as the design results.

2. Design Process

2.1 Previous Work

The first-generation diesel particulate generator for the Aerogel lab came from an independent study, again, done by Jacob Cetnar last spring. After testing the plausibility that aerogels could be used as filters, Cetnar continued his research to find a method of testing them in a scaled down environment. Inspiration for this design came from Cambustion's *DPG* in the sense that, diesel soot produced by a flame was forced through tubing using compressed air. Figure is the schematic for Cambustion's design. Essentially, diesel soot is produced from diesel fuel ignited in a burner. This soot is then pushed through the system by filtered compressed air. Cetnar's design followed this same premise but was largely scaled down [14].

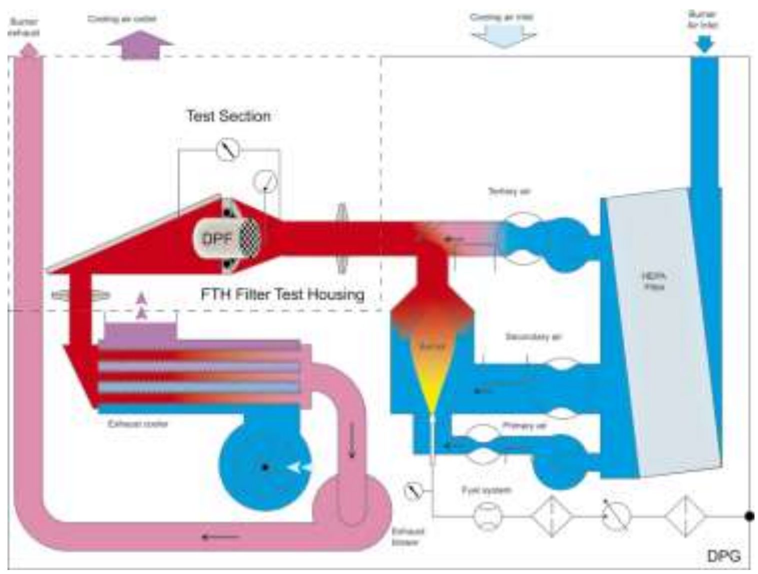

Figure 8 Schematic of Cambustions DPG [9].

An alcohol burner, filled with diesel fuel, was used as the soot source in Cetnar's design. From there, the soot traveled up the lower tubing and into the upper tubing. Once in the upper tubing, inlet air pushed the soot while a vacuum pulled it towards the test section.

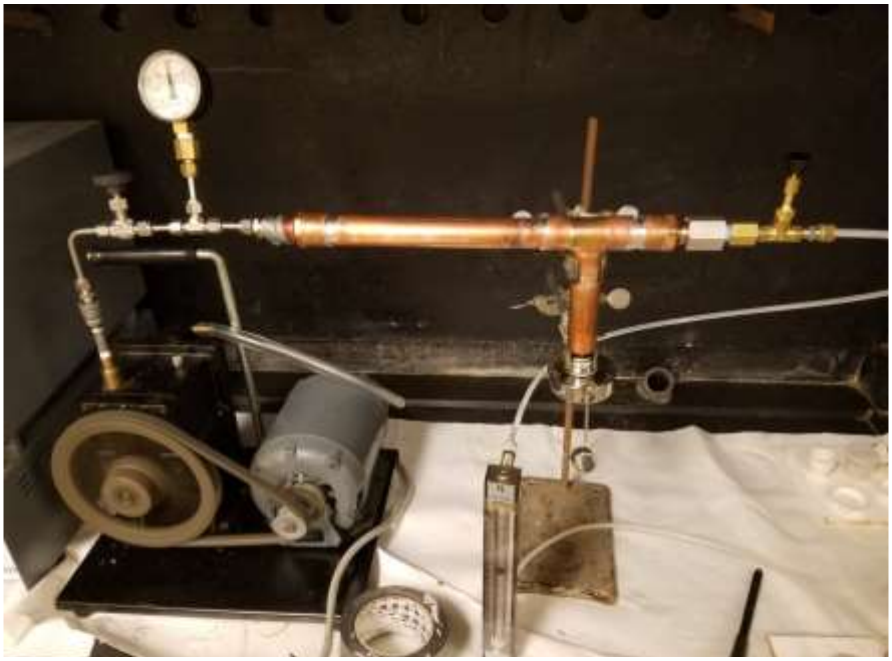

Figure 9 Cetnar's diesel particulate generator [14].

Figure 9 is the set-up that Jacob used to load aerogels with diesel soot.

After running initial tests, the design seemed to work but problems were prevalent. Among these problems was clogging. Since diesel soot is very sticky material it is hard to keep it from sticking to the sides of the piping.

Figure 10 A small layer of soot buildup on the interior of the copper piping [17].

Figure 10 shows this soot build up on the interior of the piping. After about 15 minutes of testing there was a buildup of 1/16" of soot in the pipe [14]. While this does not seem like too much, it is enough to significantly change the results of the experiment. If soot is building up on the tube then that means that not all the soot generated is reaching the aerogel meaning that it is not acting as a filter to all of the soot which has the potential to skew the results. An additional issue was that the system would heat up a lot and would eventually get to the point where it had to cool down before being used again. This does not allow for rapid or very repeatable testing, two important factors. Lastly, and the most important issue with this design is the fact that it uses a flame. While at first glance, the use of actual diesel combustion to produce diesel soot is an advantage, however, when looking at what the goal of a small-scale DPG having a flame is detrimental. Basically, what the flame does is limit the repeatability of the experiment which is essential. The aerogel needs to be loaded with soot under the same conditions each time and if there is a different flame (i.e. different conditions) each test then comparing the results will not be useful, rendering the DPG almost obsolete.

2.2 Further Research: Additional Soot Loading Method

An alternative method was presented in the paper, *Diesel Particulate Filter Test Methods*, written by Robert Locker, N. Gunasekaran and Constance Sawyer, which describes "laboratory techniques that approximate engine exposure

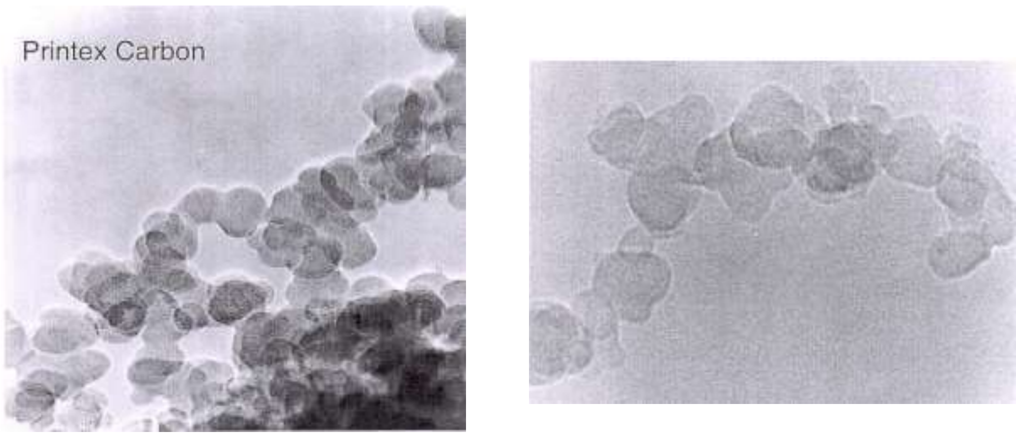

Figure 11 TEM imaging (150x) of Printex-U (left) and Diesel Soot (right) [15].

conditions." The intriguing aspect of this design is that an artificial soot is used rather than actual soot from diesel fuel [15]. Because the design revolves around the use of artificial soot, extensive research was done to determine if the artificial soot was able to adequately simulate real soot. Locker et al. chose Printex-U powder manufactured by Degussa AG which is a carbon based black powder generally used in printing ink applications [15]. After analysis, Locker et al. determined that the Printex-U, although not perfect, is a suitable replacement for diesel soot.

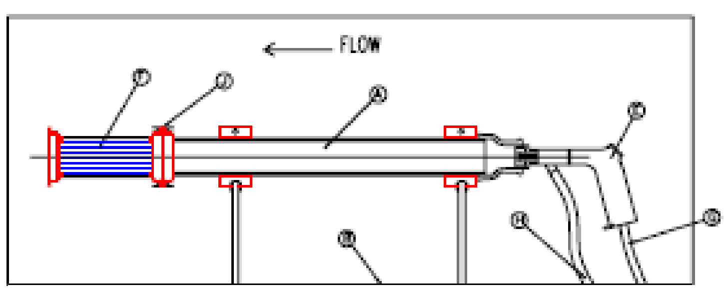

Figure 12 Basic design drawing of Locker et al.'s design [15].

Figure 11 above are two TEM images taken by Locker's team. On the left is a TEM image of Printex-U and the right is diesel soot. Due to the similar structure of both it can be determined that they hold enough similar qualities to use Printex-U as an artificial soot [15].

Another aspect of the design includes using a sandblasting gun and a screw feeder to blow the “soot” particles through the system and into the filter. By doing this, they were able to regulate the volumetric air flow rate to 20 cubic feet per minute as well as regulate the amount of Printex-U going into the system to 0.1 grams per minute [15]. Overall, due to the simple and elegant nature of this design, it is very controllable and repeatable, two very important aspects that posed problems in the previous design describe in Section 2.1.

2.3 Final Design Choice

After this initial research, it was determined that the diesel particulate generator was to be adapted from either the designs of Cetnar or Locker’s et al.’s.

The 4 characteristics examined for each design were repeatability, how realistic of an experiment, ease of manufacture, and rate of success. For repeatability, it was determined that Cetnar’s design was not as repeatable as Locker’s. This is simply because Cetnar uses a flame which, as previously mentioned, is not a very repeatable action. Whereas, the constant compressed air stream in Locker’s design is a very repeatable action. Since Cetnar, used actual diesel soot his design was very realistic while Locker used a carbon black powder. For the most realistic experiments, diesel soot is the ideal substance to be used, giving Cetnar’s design an edge. Ease of manufacture was not a large contributor but something that was taken into account. For Locker’s design it was just a long section of tubing with two connections; one for the filter housing and the other for the sandblaster, both of which were just clamped on. While Cetnar's design consisted of a connection for the compressed air, the vacuum and the lower tubing that housed the diesel flame. All in all, Locker's design was simpler to put together and thus would allow for easier troubleshooting and any necessary repairs. Lastly, the rate of success of the system was taken into account. One large problem with Cetnar's design was that it was very susceptible to clogging which could have a large impact on the results. Additionally, because of the flame the results could be different each test. Locker's design had little clogging and also used an extremely controllable air flow to blow the same amount of powder through the filter every time which increases the rate of success of the overall system.

Table 1 provides a summary of the design choices and makes clear that Locker's design has more positives.

Table 1 Design matrix for design choice.

	Locker	Cetnar
Repeatability	+	-
Reality	-	+
Ease of Manufacture	+	-
Rate of Success	+	-

With this, it was determined that the design of the scaled down diesel particulate generator will be largely based off the design of Locker et al.

2.4 Diesel Particulate Generator Design

The following sections will provide a detailed design overview of the diesel particulate generator. The design as a whole will incorporate an air gun blowing powder through 26 inches of stainless steel tubing and into a test section that will house the aerogel filter. The powder will be introduced into the system by way of a funnel/auger system.

2.4.1 Filter Test Section

A key feature of this design is that the test section is interchangeable with Union College's Catalytic Testbed (UCAT). The UCAT system is used to perform tests on catalytic aerogels by passing a mixture of gases through them. Since the UCAT system can heat aerogels up to high temperatures, and since the diesel filter test section being described can fit into the UCAT, the UCAT will be able to be used to provide heating and gas flows needed to test regeneration of the aerogel.

Tyler Gurian Union College '16 provided detailed drawings for this test when he first designed it [16]. The design of the test section contains 2 parts. The first is the main part where the aerogel will be housed, as shown in figure 13.

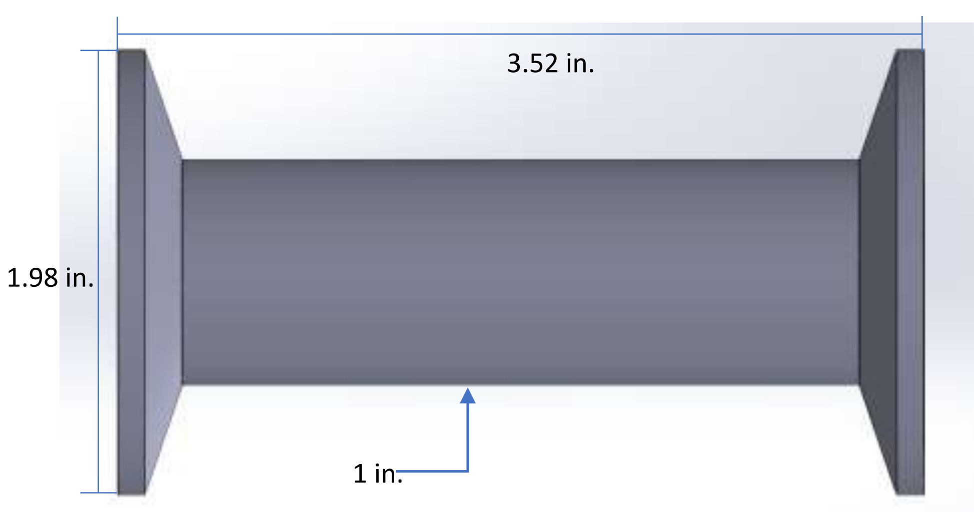

Figure 13 SolidWorks model of the main part of the test section.

Figure 14 Mesh part for DPG test cell.

Additionally, there is a 0.87-inch diameter cylinder that sticks out about 0.5 inches on the right side of the test section. This is to ensure that there is no air leaking due to the connections between tubing. The test section also utilizes two end caps with mesh on one side of them, shown below in Figure 14. These end caps have an outer diameter of 0.87 inches, and an inner diameter of 0.66 inches. The height of each cap is 0.74 inches and the mesh is tack welded to one side of the cap.

The purpose of these is to hold the aerogel in place. Ultimately, when aerogel testing occurs, the aerogel will be in granular form, thus it is necessary for something to be there in order to keep the aerogels from blowing out of the system. The original design for this part had a cross-hair metal component to support the mesh [16], however, this component could act as a soot 'stopper.' The decision was made to use the coarser, sturdier mesh, pictured above, rather than use the metal cross-hairs.

2.4.2 Air Flow Requirements

In Locker et al. the filters being tested were full scale, and so the flow rates were much higher (20 ft^3/m (cfm)) that what was required for the current work [15]. Since the DPG that is being designed is not for full scale testing, Locker's et al. experiment must be scaled down. In particular, the space velocity in the small scale test cell of the current design must match the space velocity in real DPF's.

Space velocity is defined as the inverse of the residence time (where residence time is the average amount of time a fluid particle spends in a system) [17]. Mathematically,

$$S.V = \frac{\dot{m}}{m_{sys}} \qquad \text{Eq.1}$$

Where S.V is the space velocity, $\dot{m}$ is the mass flow rate and m_{sys} is the mass of the gas in the system. Next, the space velocity of Locker's design was determined. The mass flow rate is:

$$\dot{m} = \dot{V}\rho_{air} \qquad \text{Eq. 2}$$

The volumetric flow rate of 20 cfm, specified in Locker et al. is typical of a full scale DPF and translates to 0.009439 m^3/s and the density of air at standard pressure is 1.225 kg/m^3, making the mass flow rate of Locker's system 0.01156 kg/s. The next step was to determine the mass of air contained in the DPF.

$$m_{sys} = \rho_{air}V \qquad \text{Eq. 3}$$

Where the volume of the test section was just the volume of a cylinder or,

$$V = \pi r^2 l \qquad \text{Eq. 4}$$

For Locker's system, the radius was 0.0254 m and the length was 0.1524 m [15] making the volume 0.000308 m^3. Multiplying this by the density of air produces a total mass of the air within the test cell to be 0.000379 kg. Using this, the mass flow rate and equation 1, the space velocity within the test cell of Locker's design was found to be 30.56 L/s. So space velocity in the new test section would have to be 30.56 L/s.

Instead of solving for the space velocity for the DPG design, the volumetric flow rate was solved for. Simplifying equation 1 yields;

$$S.V = \frac{\dot{V}}{V_{sys}} \quad \text{Eq. 5}$$

Where the volume of the system is also found using equation 4, with the radius of the test section as 0.0127 m and the length as 0.088 m, making the volume 0.0000459 m^3. Multiplying this by the space velocity of 30.56 1/s yielded a volumetric flow rate of 0.00136 m^3/s which is equivalent to 2.88 cfm. This is an important design parameter because this means that for the system an air gun capable of blowing air at 3 cfm will be needed. Furthermore, this value will also provide a Reynolds number from which the length of tubing before the test cell can be determined.

2.4.3 Powder Choice

The next step of the design was to either confirm that Printex-U was a suitable simulant for diesel soot or determine if another powder should be used. Referring

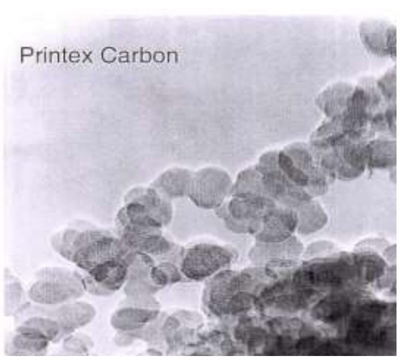

Figure 15 TEM image of Printex-U, 150x

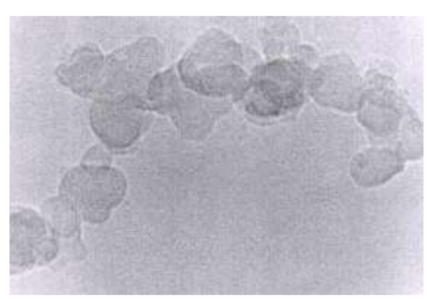

Figure 16 TEM image of diesel soot, 150x

to transmission electron microscopy (TEM) imaging (reproduced from [15] above, Figure 15 and 16) and research done by Locker et al. it was determined that Printex-U would be suitable if it proved compatible with the new, smaller scale system. However, no supplier of Printex-U was found that would ship in quantities less than one 70 pound bag, which is excessive for the needs of this system. Printex-U however, is the primary ingredient in toner cartridges for laser printers and copiers. So a used cartridge (standard HP LaserJet cartridge) was taken apart and the remaining powder was removed in order to compare with diesel soot. Additionally, upon the advice of Professor Mary Carroll in the chemistry department, another powder, lampblack pigment, was considered. An

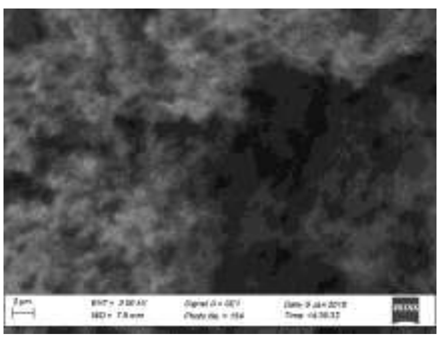

Figure 17 SEM image of lampblack pigment.

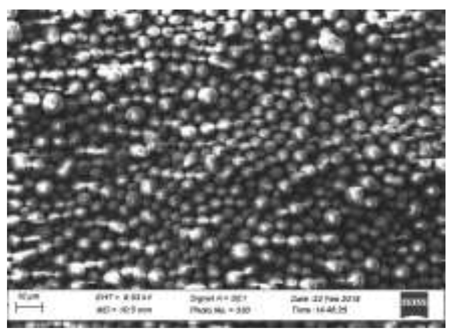

Figure 18 SEM image of the printer powder.

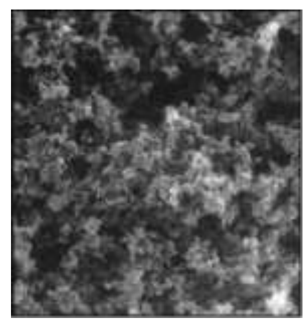

Figure 19 SEM image of diesel soot (150,000x) [23]

intriguing quality about this substance is that it has a similar oily and sticky quality just like real diesel soot, while the toner was drier. Scanning electron microscopy (SEM) imaging was conducted on the lampblack and printer powder to make comparisons to diesel soot. Looking at Figure 17 and Figure 19 they do not look identical but they both have a sort of "fluffiness" structure to them which is promising. Furthermore, combining this with the fact that lampblack has oily qualities is an added benefit. However, the printer powder, Figure 18, does not resemble either of the two samples as it is not "fluffy" at all but rather looks like small spheres. Based on the SEM images, the printer powder may not be a suitable substitute for diesel soot, but the lampblack pigment shows more promise. Based on this decision, the initial testing was done using the lampblack pigment; however, the printer powder was used in the system due to the use of Printex-U in Locker's design.

2.4.4 Air gun choice

With the CFM range found, the next step was to determine what type of air gun was to be used. Locker et al. utilized a sandblaster for their experiment; however, they were blowing the powder at 20 cfm whereas this design will only be using 3 cfm of air through the system. Therefore, a sandblaster will not work for this design.

Figure 20 Master E96 air gun purchased from TCP Global [18].

Powder paint guns are a widely available solution within the necessary cfm range. The *Master Economy E96 Single-Action External Mix Siphon Feed Airbrush Set* was selected and obtained for this work. This model flows between 2 and 5 cfm, falling perfectly within the range for this work and is also designed be used with powders [18]. After preliminary testing with the Lampblack pigment, there were significant signs of clogging in the air gun. The alterations made to the gun are described in Section 2.5.

2.4.5 Length before test cell

An important aspect of the design requires that the flow containing the particles going into the aerogel needs to be fully developed and turbulent. A fully developed, turbulent flow would ensure that the aerogel would be evenly loaded with soot. The design of this system can encourage a fully developed turbulent flow in a couple of ways. First the diameter of tubing leading up the test section can be altered; however, since the test section diameter is already set at 1" it makes the most sense to keep the rest of the tubing to a 1" diameter. Another way of ensuring the flow is fully developed and turbulent is to increase the length of the tubing. To determine this length, the Reynolds number can be calculated using the volumetric flow rate found above.

$$Re = \frac{4Q}{\pi D v} \quad \text{Eq. 6}$$

Where Q is the volumetric flow rate, D is the diameter and v is the kinematic viscosity of air. For a system with a Q of 3 cfm and a D of 1", the Reynold's number is about 4700 which is high enough to be considered a turbulent flow. This means that the following empirical relationship can be used to determine the length of tubing needed for the flow to also become fully developed.

$$L = 25D \quad \text{Eq. 7}$$

Solving for L yields a value of 25 inches. Therefore, the amount of tubing needed before the test section is at least 25 inches.

2.5 Initial Design/Testing

Figure 21 Air gun set up with 3D printed funnel and tubing attachment.

The initial design consisted of a lofted funnel being inserted into the brass piece of the air gun as shown in Figure 21. The soot simulant would be poured into the funnel and let gravity pull it into the air stream. This however, proved ineffective. The powder was going through the funnel but it was not being introduced into the air stream because there was significant clogging in the brass fitting. To alleviate the problem the brass fitting was removed entirely from the gun and was replaced with the lofted funnel, as shown in Figure 22. Improvements in the powder entering the air stream were seen, however, there was still clogging in the funnel. This clogging seemed to be alleviated by agitation to the funnel and the powder. Based on this observation it was determined that some sort of agitation device, either an auger or vibrating motor, would be necessary to move the powder through the funnel and into the air stream.

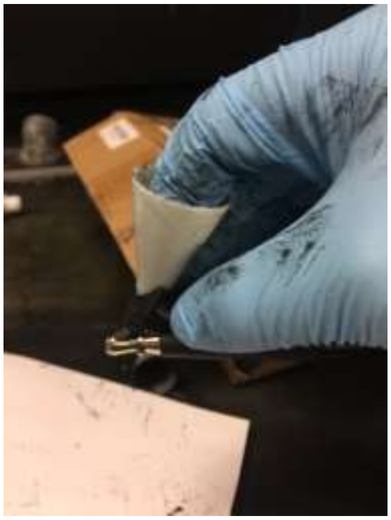

Figure 22 Air gun with brass part removed and funnel attached.

2.5.1 Auger/Funnel Design

To alleviate the clogging occurring in the funnel an auger was designed. The auger would also ensure the soot substitute would be deposited into the airstream in a controlled, constant manner. A SolidWorks model of the auger can be seen in Figure 23. The auger is just over 3 inches long and the fins are 0.10 inches wide and taper down to 0.02 inches at the bottom of the auger (a detailed drawing can be found in Appendix C). To accommodate for the auger, a straight funnel was designed, and 3-D printed. To rotate the auger, a small 3V DC motor in combination with a gear box was used. The gear box has a gear ratio of 344.2:1, reducing the angular velocity of the shaft to 38 revolutions per minute. The auger

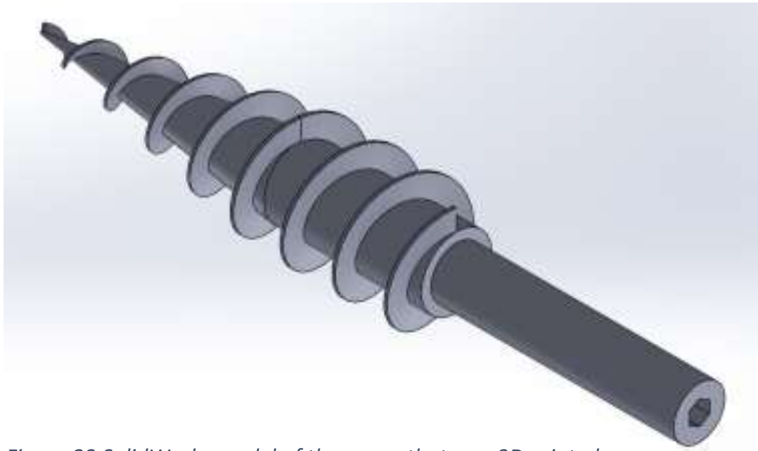

Figure 23 SolidWorks model of the auger that was 3D printed.

Figure 24 Picture of the auger attached to the gear train shaft. Auger is press fit on with tape put around for extra measure.

was then attached to the shaft of the gear box by being press fit around the shaft as seen in Figure 24.

2.5.2 Gun, Funnel/Auger Connection to Tubing

The powder deposit system needs to be connected to the rest of the device. Using SolidWorks, a part was designed that would fit snuggly around the gun tip and funnel. Furthermore, the outer diameter of the part is 0.86 inches, allowing for a tight fit inside the tubing.

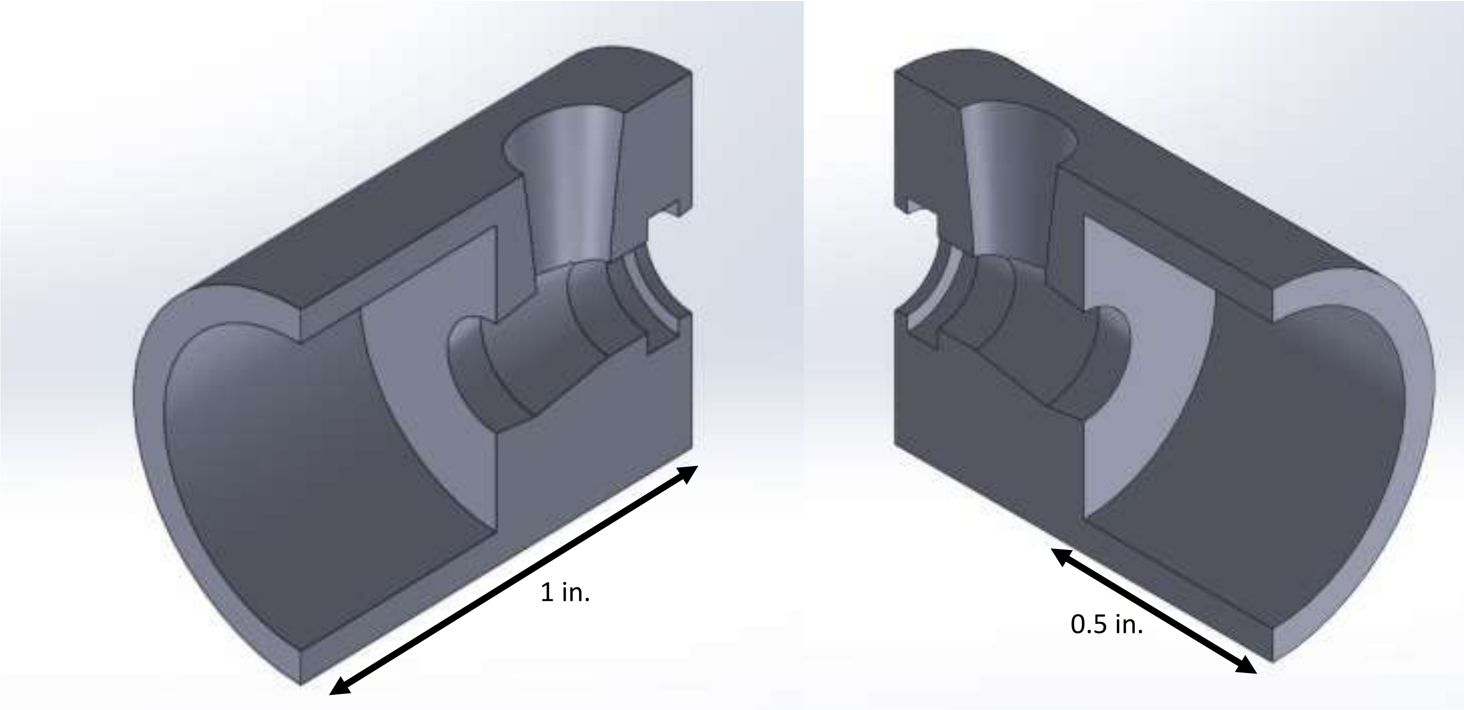

Figure 25 Connection for the funnel, auger and air gun to the rest of the system. Outer diameter of the part is 0.86 inches. This part was 3D printed out of ABS plastic.

Each half closes in around the gun and funnel holding them tightly in place. It is not, however, a permanent connection, so the gun can be taken out for proper cleaning or adjustments. Additionally, the auger slides right into the funnel and is held at the proper height by a ring stand. A cross-section view of this set up can be seen in Figure 26.

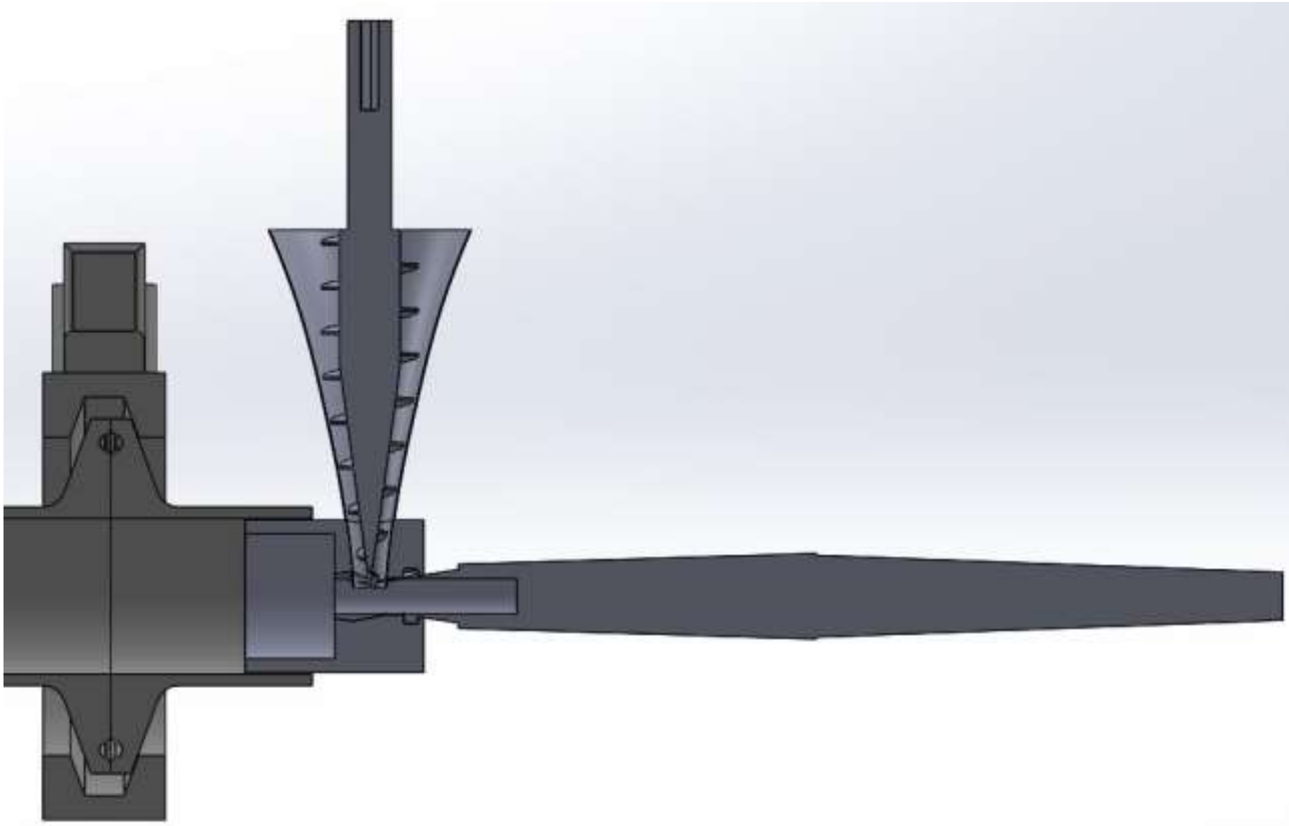

Figure 26 Cross-section view of the gun, auger, and funnel connection to the rest of the system.

2.6 Final Design

The final design of the diesel particulate generator has a Master E96 air gun blowing up to 3 cfm of air and lampblack pigment through 26 inches of tubing with a 1-inch diameter before entering the test section which houses the aerogel. The tubing is 316L Stainless Steel throughout and is part of a quick clamp system. This means that each section of tubing has flanges on it that allow for easy connections using a quick clamp. Additionally, in between each flange is a copper washer to prevent leakages. Furthermore, the 26-inch entry tubing consists of two tubes, one 18 inches and one 8 inches, and is connected by a quick clamp. A complete Bill of Materials (BOM) as well as SolidWorks drawings and files can be found in Appendix B and Appendix C, respectively, as well as the Online Appendix (link given in Appendix C). The funnel, auger, and piece connecting the gun system to the tubing were all 3D printed out of ABS plastic. Furthermore, the gear box used had a gear ratio of 344.2:1 reducing the angular velocity of the motor to 38 revolutions per minute.

Figure 27 SolidWorks model of the full DPG

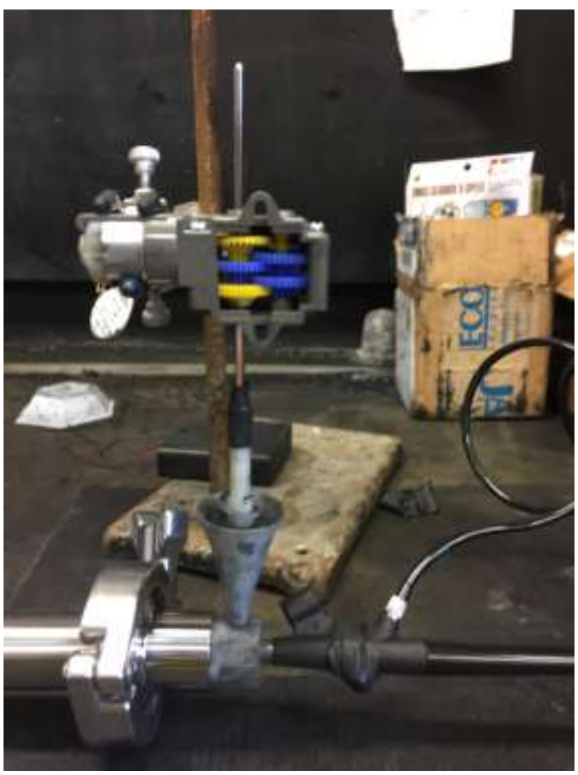

Figure 28 Picture of the auger, funnel and gun connections.

Figure 29 Picture looking down the funnel.

Figure 30 Picture of the full manufactured diesel particulate generator. The air gun is shown to the right while the test section is all the way to the left.

3. Testing

3.1 Testing without Soot Generation

Once the diesel particulate generator was manufactured initial testing without soot generation was attempted to see if any aerogel was being lost just due to the airflow.

3.1.1 Methods

To run these tests, a weighing boat was massed and then the scale was zeroed. After that roughly 0.35 grams of aerogel was measured out. One end cap was put into the test section with its screen facing the front of the test section. The aerogel was then poured into the test section and the second end cap was put in with its screen facing the back of the test section. The test section was then clamped into the system and the air was turned all the way on for 60 seconds. Once done, the test section was removed from the system. Another weighing boat was then massed and the scale zeroed again. The contents of the test section were then dumped inside the weighing boat. The end caps were then taken out of the boat and any excess aerogel remaining on the end caps was brushed off into the weighing boat. The aerogel was then massed again and those values recorded. This procedure was done 3 times.

3.1.2 Results

The results of these trials were that on average, 0.003 grams of aerogel was lost during each trial, a fairly insignificant amount that can be minimized with careful loading and unloading of the aerogel.

Table 2 Results of testing without soot generation.

Aerogel In (g)	Aerogel Out (g)	Aerogel Lost (g)	Percent Lost (%)
0.351	0.345	0.006	1.709
0.345	0.3422	0.0028	0.812
0.3422	0.3418	0.0004	0.117
	Avg. Lost (g)	0.0031	0.8793

3.1.3 Discussion

With an average loss of 0.8793 % of aerogels, it was determined that minimal aerogel was leaving the system due to air. Furthermore, the aerogel could be getting lost in the loading and unloading of the aerogel into the test section. With this in mind, it was determined that this would not affect the tests with soot generation.

3.2 Testing with Lampblack Pigment

Once it was determined that aerogel was not leaving the system due to the compressed air, soot generation was incorporated into the system.

3.2.1 Methods

Figure 31 Aerogel granules before being loaded with soot.

Aerogel was measured out in the same manner as described in Section 3.1.1. Once complete, the lampblack pigment was measured. Again, a weighing boat was massed and then the scale zeroed. Following that, lampblack pigment was spooned into the weighing boat. The amount put in varied between tests. Once the desired amount was achieved, the system, the auger and air, was turned on (Table 3 provides the masses of the aerogels and simulant that were used for each trial of testing). The lampblack was then spooned into the funnel/auger and dispersed through the tubing and into the aerogel granules in the test section. Once all of the simulant was emptied out of the weighing boat, the auger was turned off and air only ran through the system for about a minute to ensure all of the powder made it to the aerogel. The air was then turned off and the test section removed. A weighing boat was massed, and the scale zeroed, and then the contents of the test section were dumped into the boat. The end caps were removed and any residual aerogel/simulant was brushed off into the weighing boat. The boat was then massed again and the data recorded.

3.2.2 Results

Table 3 contains the different results with different loading conditions.

Table 3 Results of tests with the lampblack pigment.

Trial	Powder Type	Aerogel In (g)	Soot In (g)	Aerogel Out (g)	Soot Captured (g)	Weight Increase (%)	Soot Captured (%)
1	Lampblack	0.1415	1.1213	0.1643	0.0228	16.11	2.03
2	Lampblack	0.2881	0.8163	0.3247	0.0366	12.70	4.48
3	Lampblack	0.4679	1.1856	0.5178	0.0499	10.66	4.21
4	Lampblack	0.1944	0.5401	0.212	0.0176	9.05	3.26
				Avg.	0.0317	12.13	3.50

As shown in the chart, aerogel does collect the lampblack thus does act as filter for soot. However, it only captured an average of 0.0317 g or 12.13% of its mass before being loaded with soot. However, on average, only 3.50% of the soot simulant was captured by the aerogels. These numbers, although they show that aerogels are capable of filtering soot, are not nearly as good as they need to be to be effective in a diesel particulate filter.

Figure 32 Aerogel granules post testing with lampblack pigment. Notice the discoloration of the aerogel to a shade of gray.

3.2.3 Discussion

There are a few reasons as to why the numbers are so low. The first is that not all of the soot is reaching the aerogel, however, after testing, the DPG was taken apart and the inner surface of the tubing was analyzed to see how much soot stuck to the surface. Figure 33 clearly shows that there is some soot build up on the interior of the tube, however this picture was taken at the end of the four tests and was not consistent throughout the entire length of the tubing. This means that this

Figure 33 Picture of soot simulant build up on the interior of the DPG

build up is insignificant and more soot is reaching the aerogel but just passing through instead of being filtered. Furthermore, it is possible that there is not enough aerogel/soot in the system to see an appreciable amount of soot captured. Lastly, although SEM images show that lampblack and diesel soot share similar qualities, there might be more differences. For example, a particle of lampblack as a diameter of 95 nm [19] which is very large when compared with the diameter of a diesel soot particle, which falls between 25-20 nm [15]. This fact alone could explain why aerogels do not capture the lampblack due to the porosity of the silica aerogels that were being used. A more in depth discussion of this idea will occur later in the report. Additionally, the aerogel did experience a change in coloration; however it was not too severe, but still provided evidence of soot being loaded;

Figures 31 and 32 demonstrate the difference in aerogel coloration prior to, and after testing, respectively.

3.3 Testing with the Printer Powder

Although it was deemed that the lampblack pigment was a suitable diesel soot simulant, the results from the initial testing left a lot more questions. Although, the lampblack looked and acted like diesel soot, the results were a lot different from Locker et al.'s results as well as Cetnar's results which were produced with real diesel soot. To further examine this idea, it was decided that the printer powder would be tested. The DPG was thoroughly cleaned as to avoid any contamination with the lampblack.

3.3.1 Methods

The same methods described in Sections 3.1.1 and 3.2.1 was used for the loading of aerogels and the printer powder.

3.3.2 Results

Table 4 provides the results of the printer powder tests.

Table 4 Results from trials with the printer powder.

Powder Type	Aerogel In (g)	Soot In (g)	Aerogel Out (g)	Soot Captured (g)	Weight Increase (%)
Printer Powder	0.2854	1.4807	0.4011	0.1157	40.54
Printer Powder	0.2355	1.212	0.2963	0.0608	25.82
Printer Powder	0.4723	1.1772	0.5624	0.0901	19.08
			Avg.	0.0889	28.48

As shown, the aerogel seems to capture the printer powder better than the lampblack holding on average 28.48% of its own weight of the powder. Furthermore, the aerogels saw a significant change in coloration, turning from clear to black.

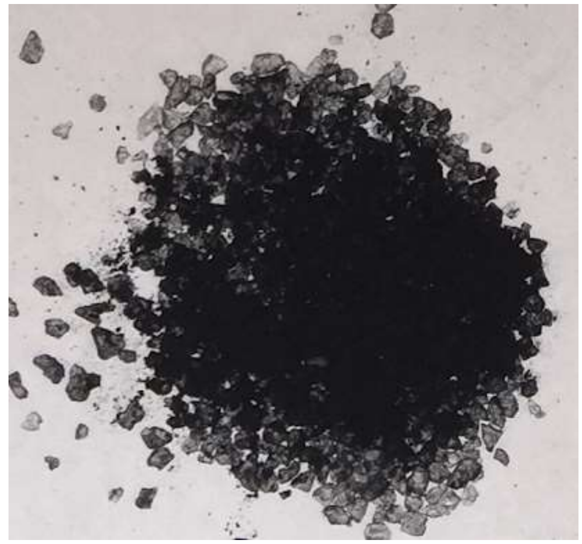

Figure 34 Aerogel granules post testing with the printer powder. Notice the black discoloration of the aerogels.

3.4 Discussion of Results

3.4.1 Discussion of Test Results

Table 5 Combined results from both the lampblack and printer powder tests.

Trial	Powder Type	Aerogel In (g)	Soot In (g)	Aerogel Out (g)	Soot Captured (g)	Weight Increase (%)	Soot Captured (%)
1	Lampblack	0.1415	1.1213	0.1643	0.0228	16.11	2.03
2	Lampblack	0.2881	0.8163	0.3247	0.0366	12.70	4.48
3	Lampblack	0.4679	1.1856	0.5178	0.0499	10.66	4.21
4	Lampblack	0.1944	0.5401	0.212	0.0176	9.05	3.26
				Avg.	0.0317	12.13	3.50
	Powder Type	**Aerogel In (g)**	**Soot In (g)**	**Aerogel Out (g)**	**Soot Captured (g)**	**Weight Increase (%)**	**Soot Captured (%)**
5	Printer Powder	0.2854	1.4807	0.4011	0.1157	40.54	7.81
6	Printer Powder	0.2355	1.212	0.2963	0.0608	25.82	5.02
7	Printer Powder	0.4723	1.1772	0.5624	0.0901	19.08	7.65
				Avg.	0.0889	28.48	6.83

Although it was determined that the lampblack was structurally a better substitute for diesel soot, the printer powder tests provided significant results. Comparing the physical appearance of the aerogels post-testing; the aerogels loaded with the printer powder physically resemble aerogels that were loaded with diesel soot in

Cetnar's experiment. Figure 35 shows five of the seven aerogels post-testing of the trials in the table above. Trials 7, 6 and 5 were all done using printer powder in all 3 cases the physical characteristics are more aligned diesel soot loading

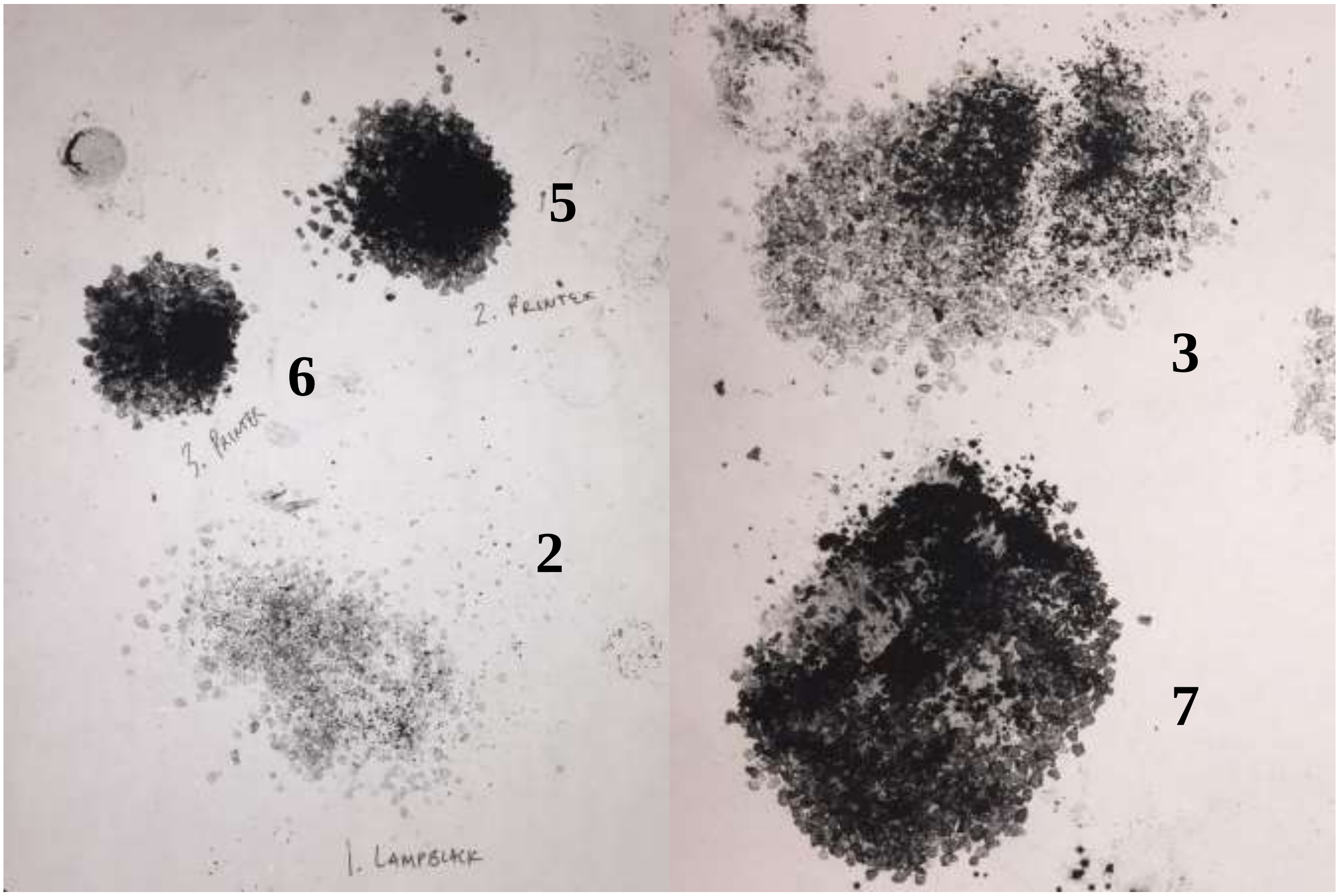

Figure 35 Photos of the aerogel post testing. The numbers in the figure correspond to the trial numbers in Table 5.

rather than trials 3 and 2 which were tests done with lampblack pigment. Additionally, the aerogels in the trials done using the lampblack the aerogels do not seem to be absorbing the powder but rather just blocking them. Whereas in the trials done using the printer powder, it appears that the aerogels are absorbing the powder rather than just blocking. Based off of this description alone it seems that the aerogels do a better job at actually filtering the printer powder rather than the lampblack.

However, another interesting difference between the two powders is how they behave in the system. The lampblack pigment becomes very smoky and resembles what would be expected to come out of a diesel exhaust. While the printer powder, barely gets smoky at all. Two tests were run without aerogel in the system; one was just the lampblack while the other was just the printer powder. Figure 36 is a still image captured from the videos taken of the tests (these videos

Figure 36 Still images captured during testing. On the left is a test of the lampblack pigment and a clear puff of smoke resembling diesel exhaust can be seen exiting the test section. On the right is a test of the printer powder where there is little to no smoke that is being generated.

can be seen in the Online Appendix (link given in Appendix C). The image on the left is of the lampblack leaving the test cell, while the image on the right is of the printer powder leaving the test cell. Again, neither test cell has aerogels in them so the powder is going through the system un-impeded. The lampblack clearly has a thick black smoke coming out of the DPG while the printer powder has a barely noticeable lighter smoke. One explanation for this is that the printer powder could be getting stuck in the DPG itself and not making it all the way to the test cell. However, the trials in Table 5 clearly show that the powder is reaching the test cell because the aerogels are capturing more of the printer powder than the lampblack.

Lastly, going back to the structural difference between the two powders could explain the differences between the two powders and their interactions with aerogels. As mentioned previously, the particle diameter size of the lampblack is roughly 95nm [19] while the particle diameter size of the printer powder, based off of SEM images, is roughly 3 µm or 3000 nm. The difference in particle size is likely the reason for the differences in filtering ability in the aerogel. Since the lampblack is much smaller in size, it could have easily gone right past the aerogels rather than actually coming in contact with the aerogel granules. Conversely, because the printer powder is much larger it may have been a lot

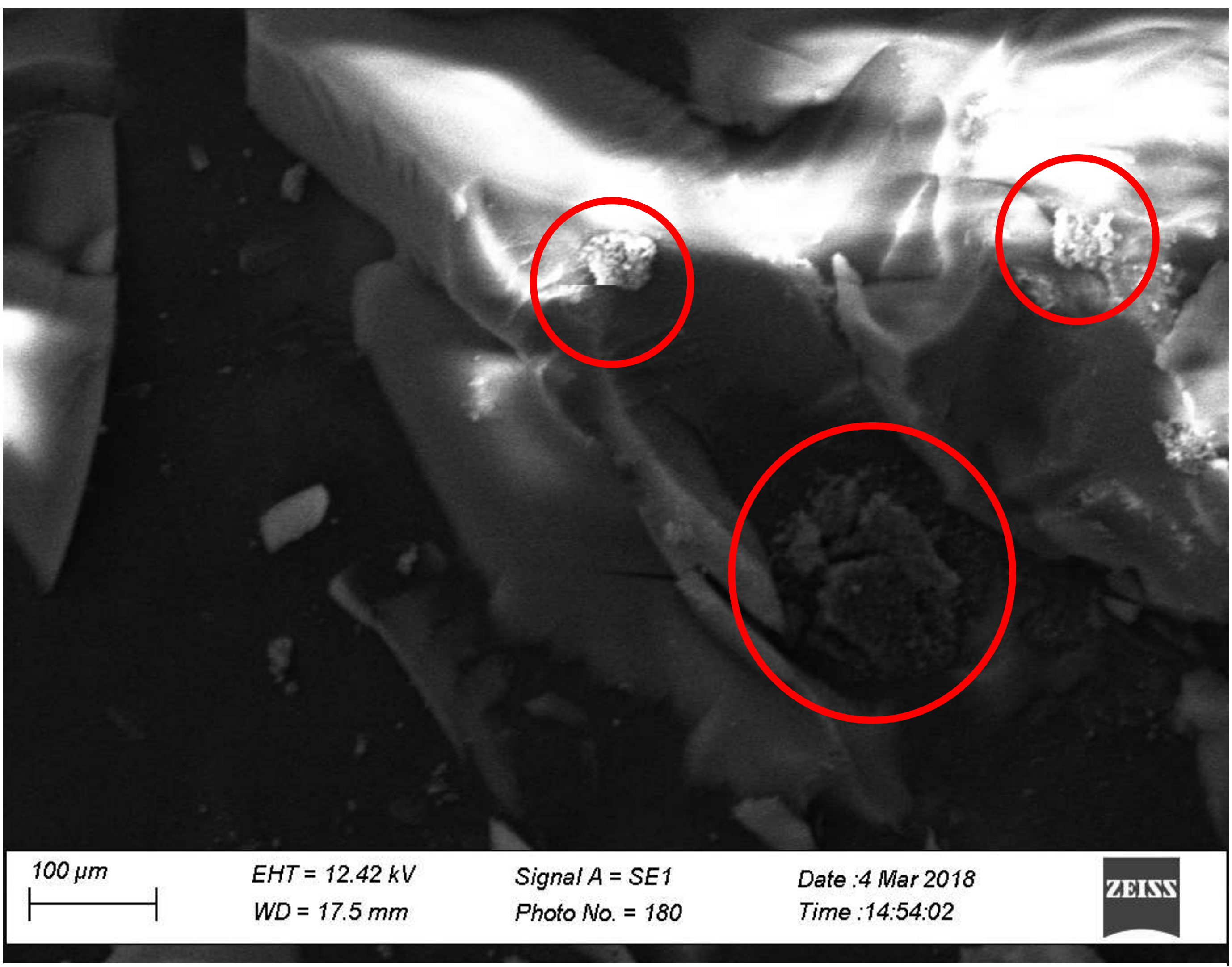

Figure 37 SEM image of the lampblack pigment loaded on the aerogel. Notice the clumps of lampblack on the aerogel.

easier for the particles to stick to the aerogels. SEM images were taken of the aerogels post-testing for each the lampblack and printer powder. Figure 37 is the SEM image of the lampblack and the aerogel. The three red circles highlight areas of large clumps of the lampblack powder have stuck to the aerogel. Whereas in Figure 38, many particles of the printer powder can be clearly seen stuck to the aerogel which might explain why the aerogel was capturing more of the printer powder. For the lampblack, if the aerogel was just capturing random clumps of the powder and completely missing the individual particles because they were too small, that may explain why the aerogel was not picking up as much lampblack as it was printer powder.

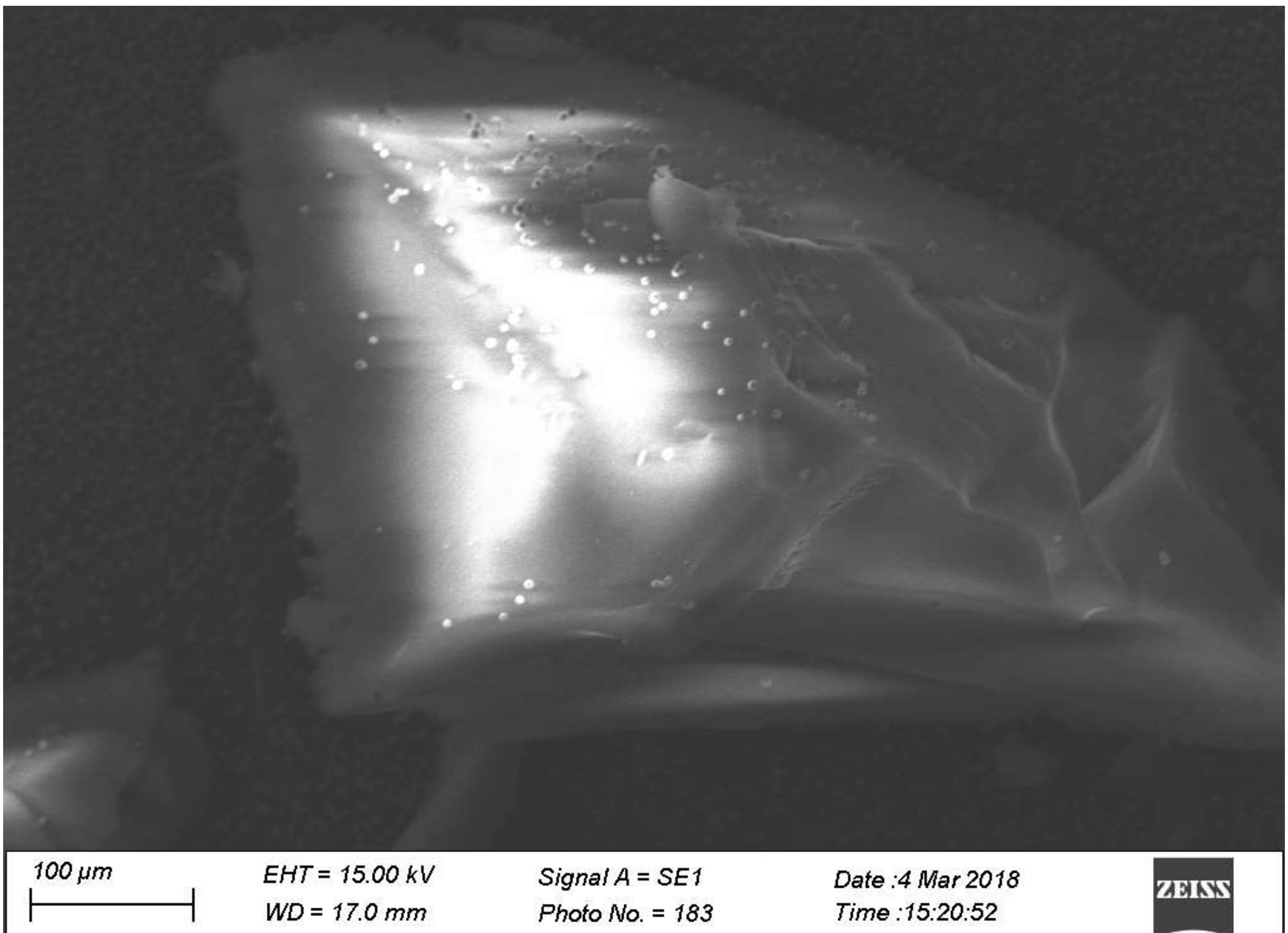

Figure 38 SEM image of the printer powder loaded on the aerogel. Notice the individual particles of the printer powder on the aerogel.

3.4.2 Discussion of Design Results

A final observation about the differences between the two powders is that regardless of how the aerogel was affected, the designed diesel particulate generator proved that it can consistently generate a diesel particulate substitute and deposit it onto aerogel. The goals of this design were to create a scaled down version of a diesel particulate generator that is capable of providing repeatable and rapid testing of aerogel filters in a small scale lab environment. Furthermore, the design was to alleviate any issues of clogging that may skew results. The results of the designed DPG demonstrate that it is capable of providing repeatable and controlled testing. The auger rotating at a constant angular velocity ensures that the powder is evenly deposited into the airstream and thusly loads the aerogel at an even rate from test to test. The use of soot simulants rather than diesel soot also proved to limit the clogging and allow for more testing without having to clean the DPG as often.

4. Conclusions and Future Work

4.1 Conclusions

The diesel particulate generator described in this report works as designed and provides a method for the Union College Aerogel lab to test aerogels as potential diesel particulate filter. The DPG provides consistent and repeatable testing that can used to gather important information about aerogels as pollutant filters. Furthermore, it was determined that the DPG can handle different types of soot simulants. The lampblack pigment appeared to resemble soot the most in the SEM images but when tested the aerogel reacted in a different way than expected. Instead of really capturing the lampblack, the aerogel seemed to just block clumps of the lampblack. Conversely, the printer powder, which did not physically resemble soot very well, reacted with the aerogel completely different. In these tests, the aerogels seem to actually absorb and filter the printer powder, which was expected to happen with the lampblack. The difference in the reaction of the aerogels is a unique finding which could shed light on how aerogels respond to different pollutants. Additionally, the different types a simulant proves that the DPG is capable of handling different materials and perhaps, provides the possibility of diversifying the DPG in the future.

4.2 Future Work

To optimize this design even further a few things could be done. Due to the methods of Locker et al. and the results found in this project, it would be interesting to try and run this test with Printex-U and see how it would differ when compared with the lampblack. Although the printer powder most likely contained some Printex-U it is highly unlikely that it was 100% Printex-U thus, it may be useful to run tests with 100% Printex-U. These tests may prove useful to gaining an understanding of how aerogels react different with different pollutants.

Additionally, a method for testing filter efficiency needs to be developed. While the current method of weighing the aerogel before and after the testing provides useful information, it does not provide nearly enough information to provide the filter efficiency of an aerogel filter. Pressure drop, temperature measurements, and a measure of the amount of particles entering and leaving the filter would be especially useful for determining the filter efficiency. Instruments such as pressure gauges and particulate meters could be helpful if implemented before and after the test cell.

Lastly, the type of aerogel filter that is being used could be modified. While the aerogel granules used do provide a quick and easy way to see what happens to aerogels in certain loading situation, they do have flaws. Loading the aerogel granules proved difficult and at times inefficient due to the small size of each granule. Furthermore, as seen with the lampblack powder, anything that has a small particle size (like diesel soot) may be able to get through the spaces in between the granules. Unless packed extremely tightly, large gaps between the granules exist. These large gaps would not be a part of a filter and therefore having them in the current filter design may provide inaccurate results. An alternative to this would be to create a mold that would allow for aerogel monoliths to be tested which would also have the added effect of aerogels porosity, an important characteristic in filtering ability.

Acknowledgements

I would like to thank Professor Bradford Bruno for taking the time to advise me through this project and for providing me with the guidance and knowledge to succeed. Also, I would like to thank Professors Ann Anderson and Mary Carroll, as well as the rest of the aerogel lab, for offering insight and opinions towards this project. I'd also like to thank Paul Thompkins and Rob Harlan in the machine shop for helping me with my parts. I'd also like to thank Stan Gorski, Duncan Mularz and Sam Kupiec for helping me out with the 3D parts that I needed throughout the course of this project. Lastly, I'd like to thank any of my engineering friends that helped me along the way.

Appendix A: Procedure

A.1 Loading Aerogel

- Remove test section from DPG by unclamping the quick-clamp
- Make sure inside of test section is clean. Use either a chemical wipe or rinse with water. If water is used, make sure to dry out test section completely.
- Insert an end cap into the test section. The mesh section of the cap should be facing the front of the test section. Refer to Figure A.1 for a visual representation of this.

Figure A.1 DPG Test Section with the front labelled.

- Zero a digital scale by using a weighing boat.
- Spoon the desired amount of aerogel onto the weighing boat and mass.
- While holding the test section vertical with the front facing down, carefully pour the measured amount of aerogel into the test section and empty all of the aerogel.
- Once all of the aerogel is in the test section, insert the second end cap into the test section. The mesh part should be facing the back.
- Add the test section back to the DPG by inserting the front end of it into the DPG tubing and then adding the quick clamp to the flanges.

A.2 Loading Soot Simulant

- Mass the soot simulant by using the same method as the aerogel massing. I.e. Zero scale, and then spoon desired amount onto the weighing boat.

A.3 Running the DPG

- Turn on the air to full volume and then turn on the battery pack to rotate the auger.
- Start pouring the powder down into the funnel at a relatively constant rate.
- Once the powder is emptied from the weighing boat, turn off the auger.

- Leave the air on for roughly one and a half minutes to make sure all soot simulant is out of the system.
- Turn air off.

A.4 Gathering Results

- Remove test section from DPG. Keep as horizontal as possible so the contents are not spilled.
- Zero the scale by using a weighing boat. Remove the weighing boat once the scale is zeroed.
- Dump the contents of the test section into the weighing boat. Carefully remove the end caps and brush off an excess aerogel/soot simulant back into the weighing boat.
- Mass the contents in the weighing boat and record values.

A.5 Important Notes

- If switching between soot simulants be sure to thoroughly clean the DPG to avoid contamination.
- After 5 trials the DPG should be cleaned.
-When cleaning, be sure to clean the interior of all of the tubing, as well as the funnel, auger, nozzle of the gun and the two pieces connecting the gun to the rest of the DPG.
- Water cleans DPG well, but all parts of the DPG must be 100% dry before tests resume.
- Re-assemble DPG as shown in the Figures A.2 and A.3

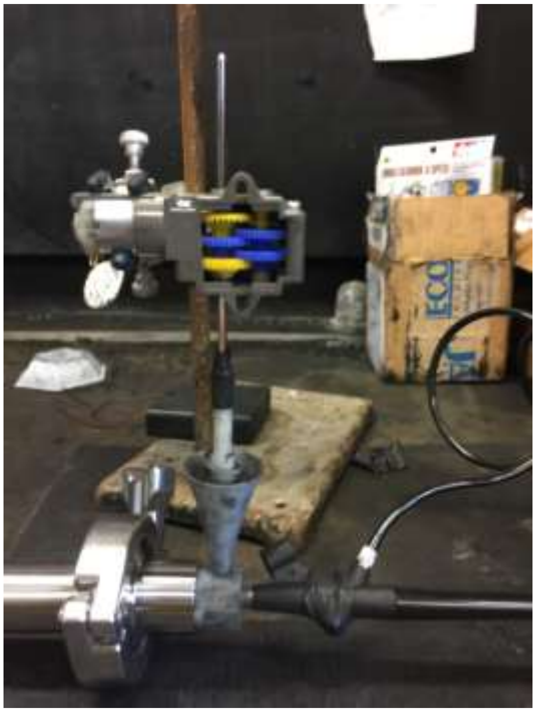

Figure A.2 Gun and Funnel/Auger

Figure A.3 Full DPG setup

Appendix B: Bill of Materials

Table B.1: Bill of Materials (Refer to Figures B.1 and B.2 for Item No. References)							
Item No.	**Item Name**	**Vendor**	**Part No.**	**Unit Price ($)**	**Qty.**	**Total Price ($)**	**Comment**
1	Air Gun	TCP Global	MAS E96	12.96	1	12.96	Air Gun used in design. Modifications specified in above report
2	Air Gun to DPG Connection	Union College 3D Printer	n/a	n/a	n/a	n/a	3D printed out of ABS plastic, SolidWorks drawings in Appendix C
3	Funnel	Union College 3D Printer	n/a	n/a	n/a	n/a	3D printed out of ABS plastic, SolidWorks drawings in Appendix C
4	Auger	Union College 3D Printer	n/a	n/a	n/a	n/a	3D printed out of ABS plastic, SolidWorks drawings in Appendix C
5	Gear Box	Union College ME Dept.	n/a	n/a	n/a	n/a	Obtain from Professor Hodgson. Used in ESC 100
6	Motor	Union College ME Dept.	n/a	n/a	n/a	n/a	Obtain from Professor Hodgson. Used in ESC 101
7	Butt-Weld Adapter	McMaster Carr	50485K161	9.60	1	9.60	316L Stainless Steel, Used to help connect gun to the rest of the DPG
8	8" Tube	Union College Aerogel Lab	n/a	n/a	n/a	n/a	Obtained from the Union College Aerogel Lab, used as length of the DPG
9	18" Tube	McMaster Carr	50485K73	140.57	1	140.57	316L Stainless Steel, majority of the length of the DPG
10	Test Section	McMaster Carr	50485K74	119.48	1	119.48	6" 316L Stainless Steel Flanged Tubing, Used as Test Section, Modifications specified in SolidWorks Drawings Appendix C
10.1	Test Section Insert	McMaster Carr	89495K225	32.26	1	32.26	12" 304 Stainless Steel Round Tube, used for the insert of the test section. Refer to Appendix C for Solidworks Drawings
11	End Cap	McMaster Carr	89495K226	n/a	n/a	n/a	Manufactured out of the 12" 304 Stainless Steel Round Tube that was used for the test section insert
11.1	Wire Mesh	Union College Aerogel Lab	n/a	n/a	n/a	n/a	Wire mesh provided by Union College Aerogel Lab, spot welded onto end caps.
12	Wing Nut Clamp	McMaster Carr	4322K152	9.44	2	18.88	Quick Clamps to hold system together, 3rd quick clamp was obtained from the Union College Aerogel Lab
13	Washers	McMaster Carr	97725A500	13.48	1	13.48	Copper washers used in between tubing flanges to prevent leakages in the system
	Lampblack Pigment	Natural Pigments	480-50	14.85	1	14.85	Lampblack pigment used as diesel soot simulant
					Total	362.08	

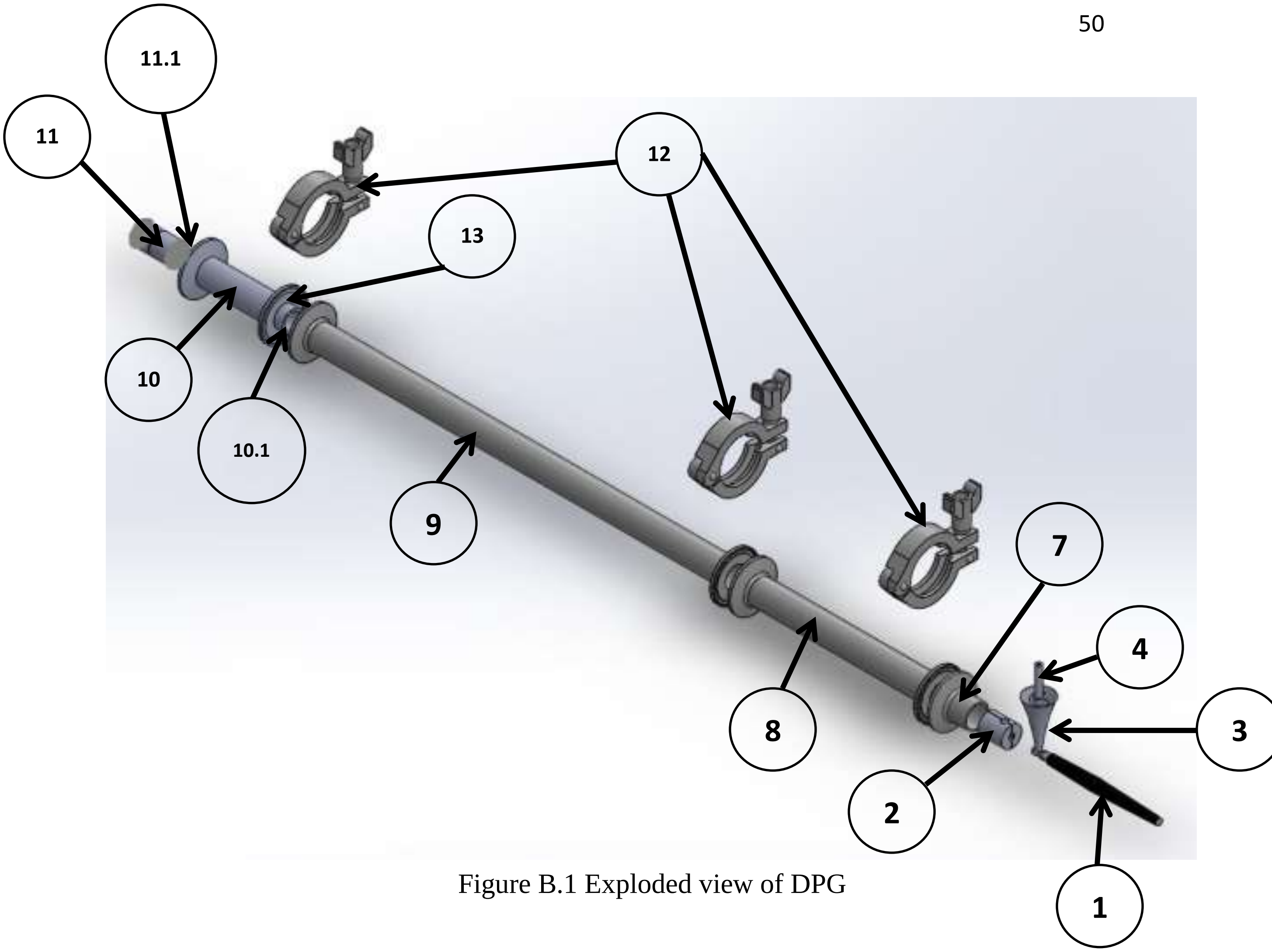

Figure B.1 Exploded view of DPG

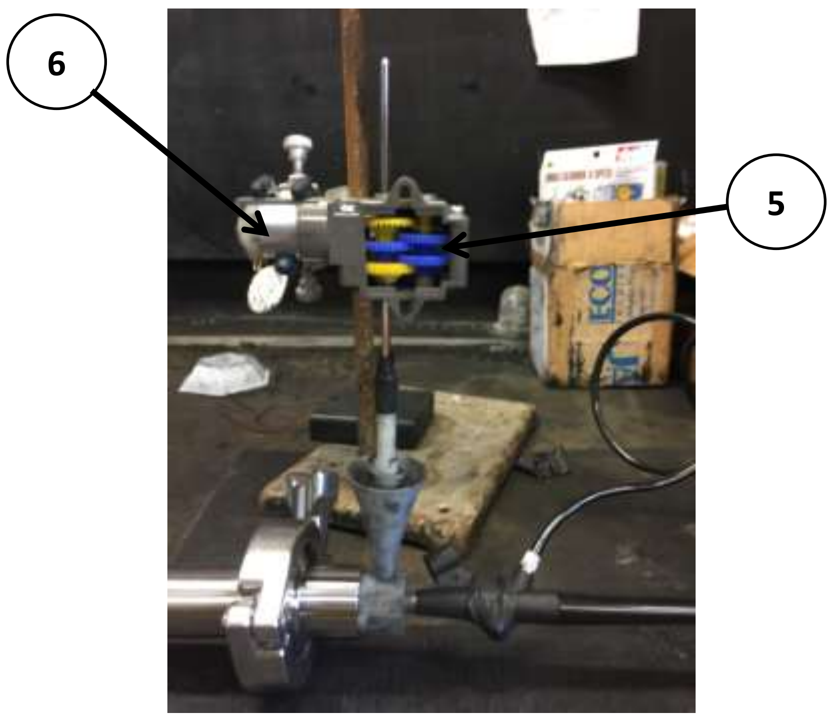

Figure B.2 Motor and Gear Box of DPG

Appendix C: SolidWorks Drawings

C.1 Test Section (Part 10)

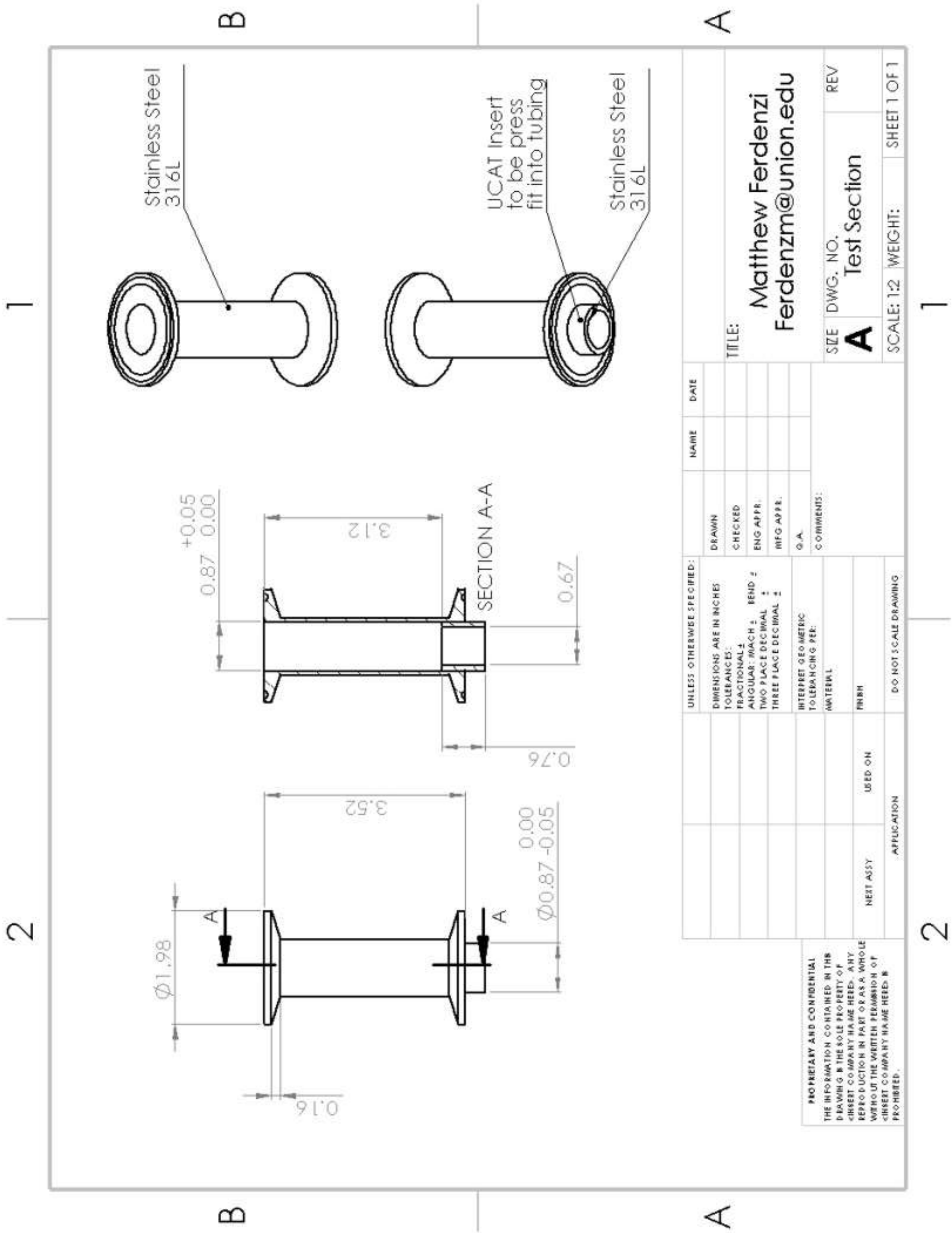

Figure C.1 SolidWorks drawing of the test section.

C.2 Test Section Insert (Part 10.1)

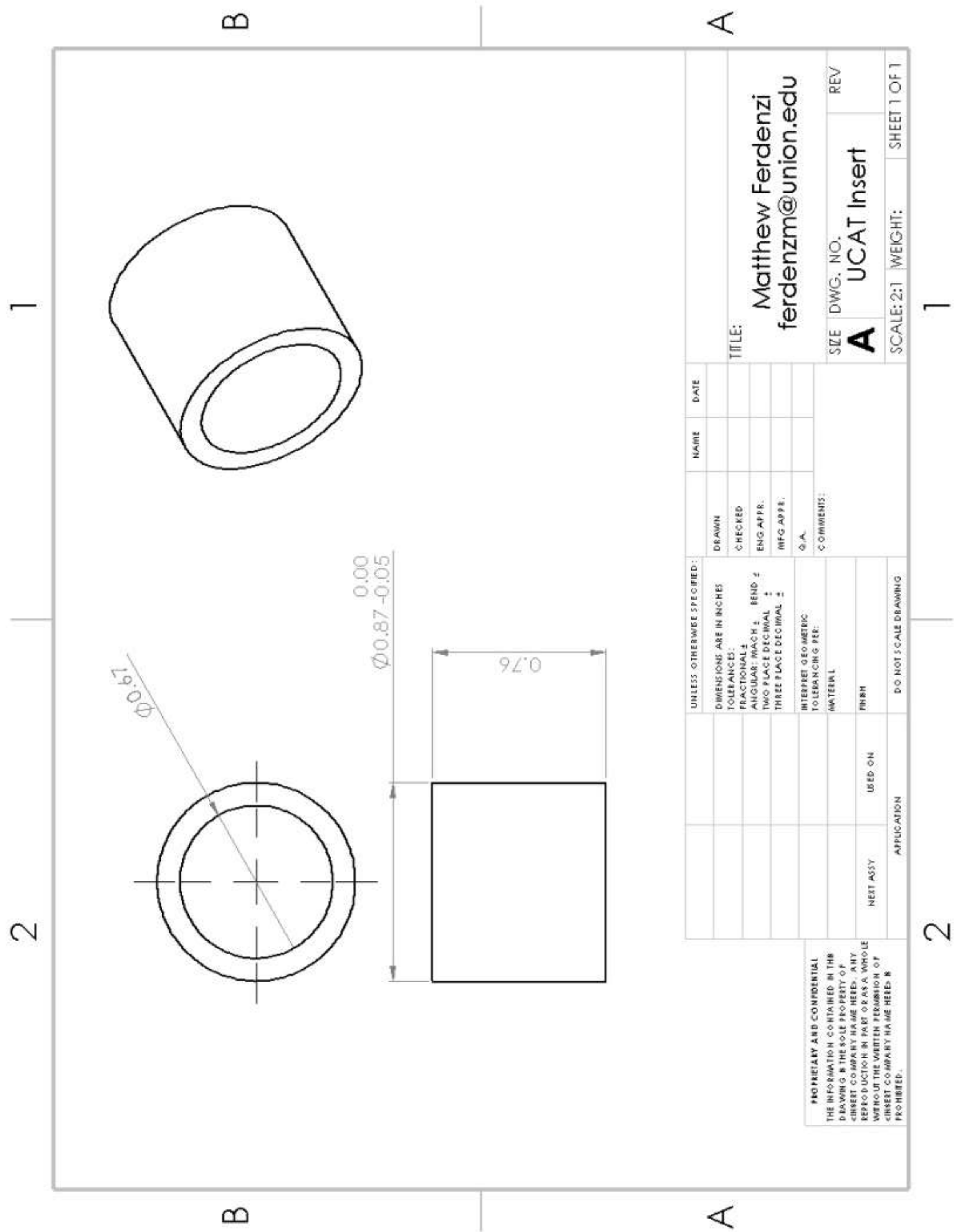

Figure C.2 SolidWorks drawing of the test section insert.

C.3 UCAT End Cap (Part 11)

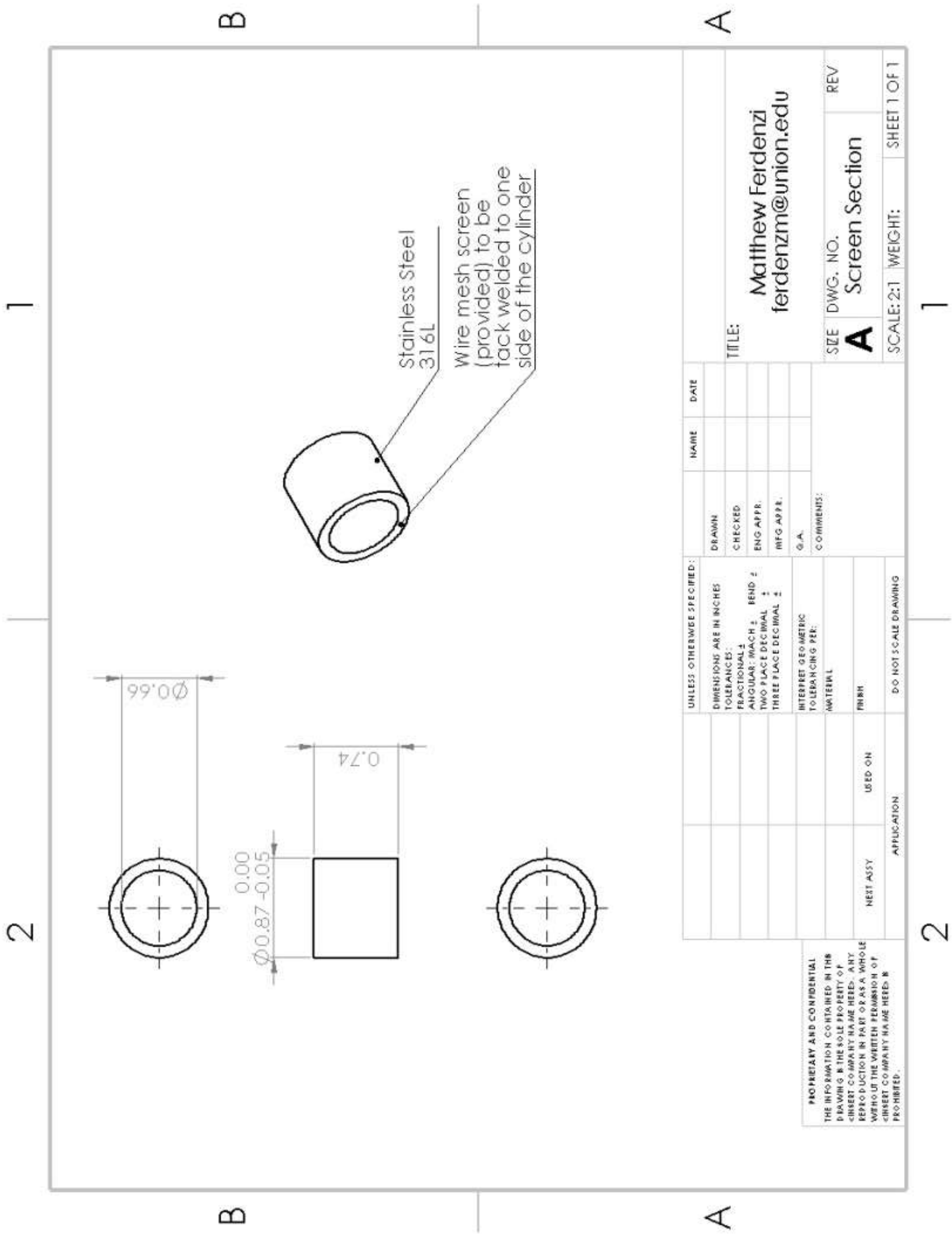

Figure C.3 SolidWorks drawing of the end cap.

C.4 Auger (Part 4)

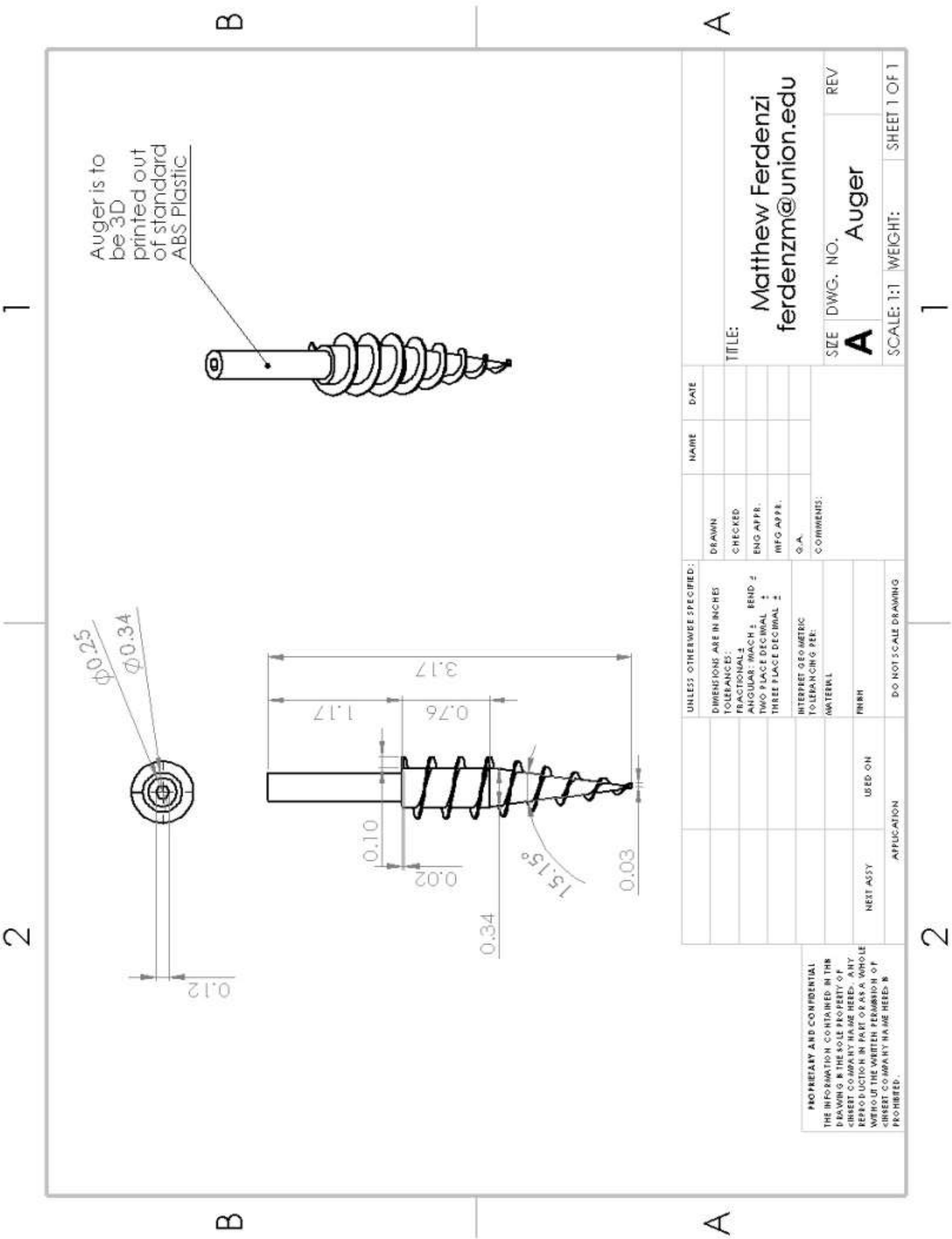

Figure C.4 SolidWorks drawing of the auger.

C.5 Funnel (Part 3)

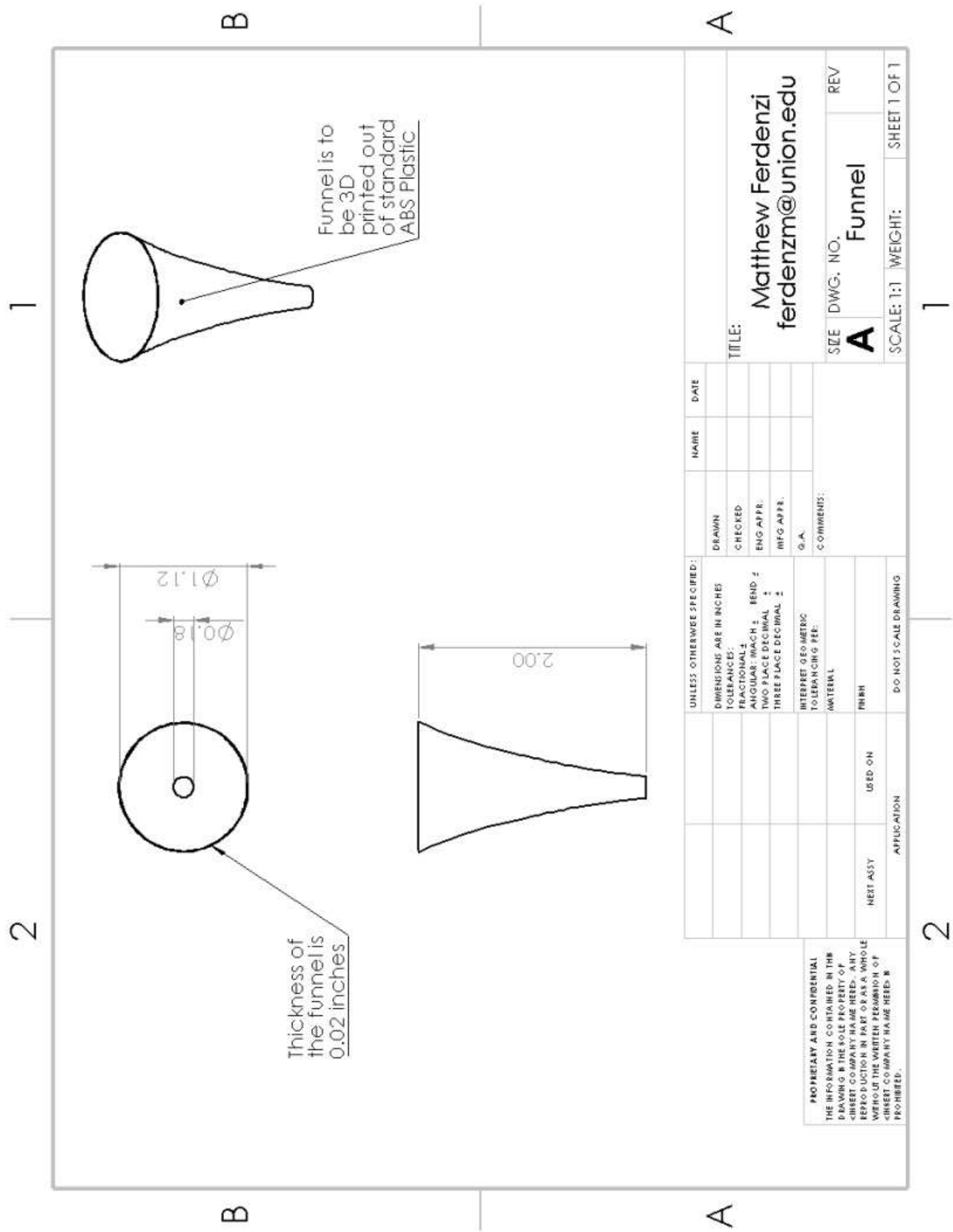

Figure C.5 SolidWorks drawing of the funnel.

C.6 Gun Connection (Left Side) (Part 2)

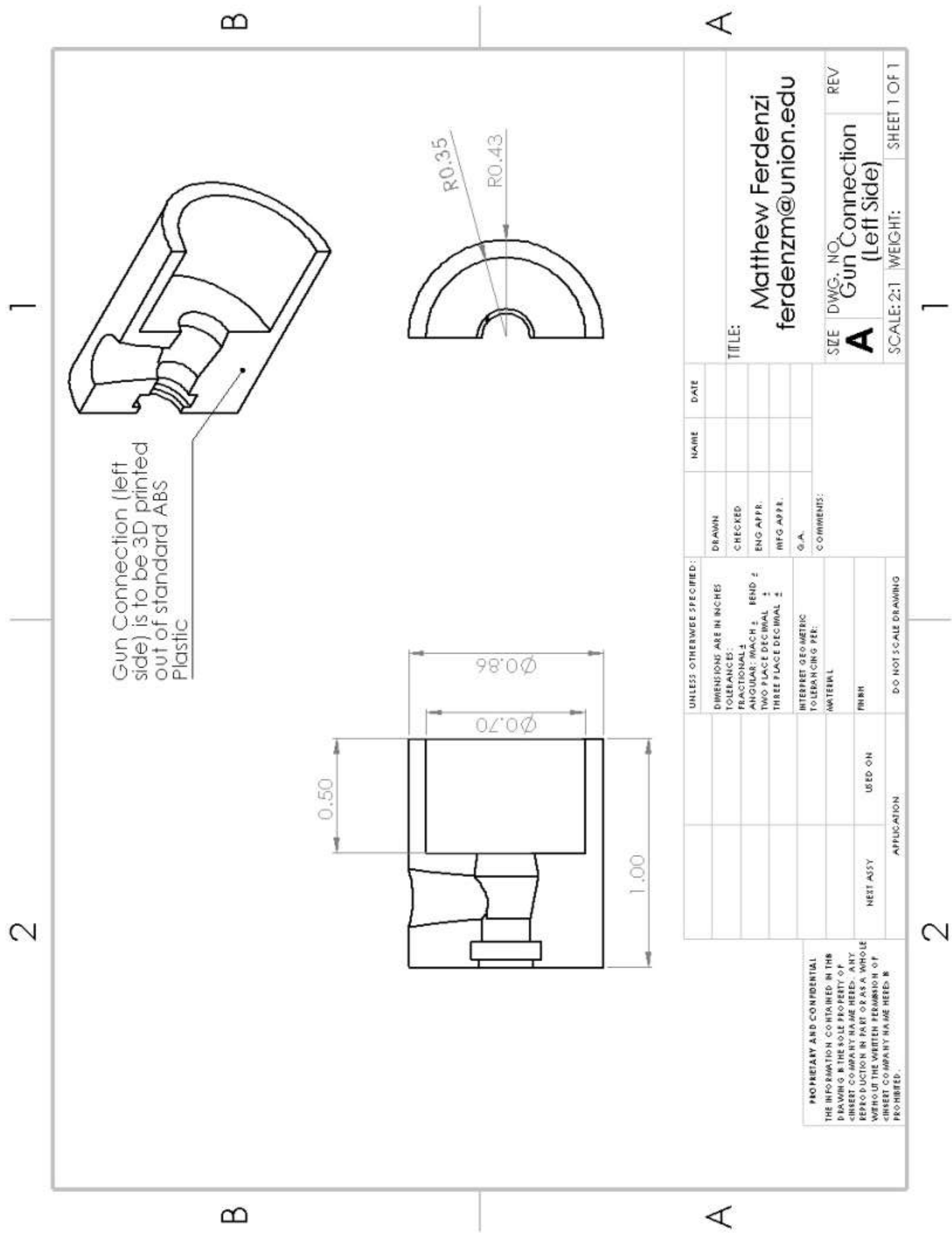

Figure C.6 SolidWorks drawing of the left side of the gun connection.

C.7 Gun Connection (Right Side) (Part 2)

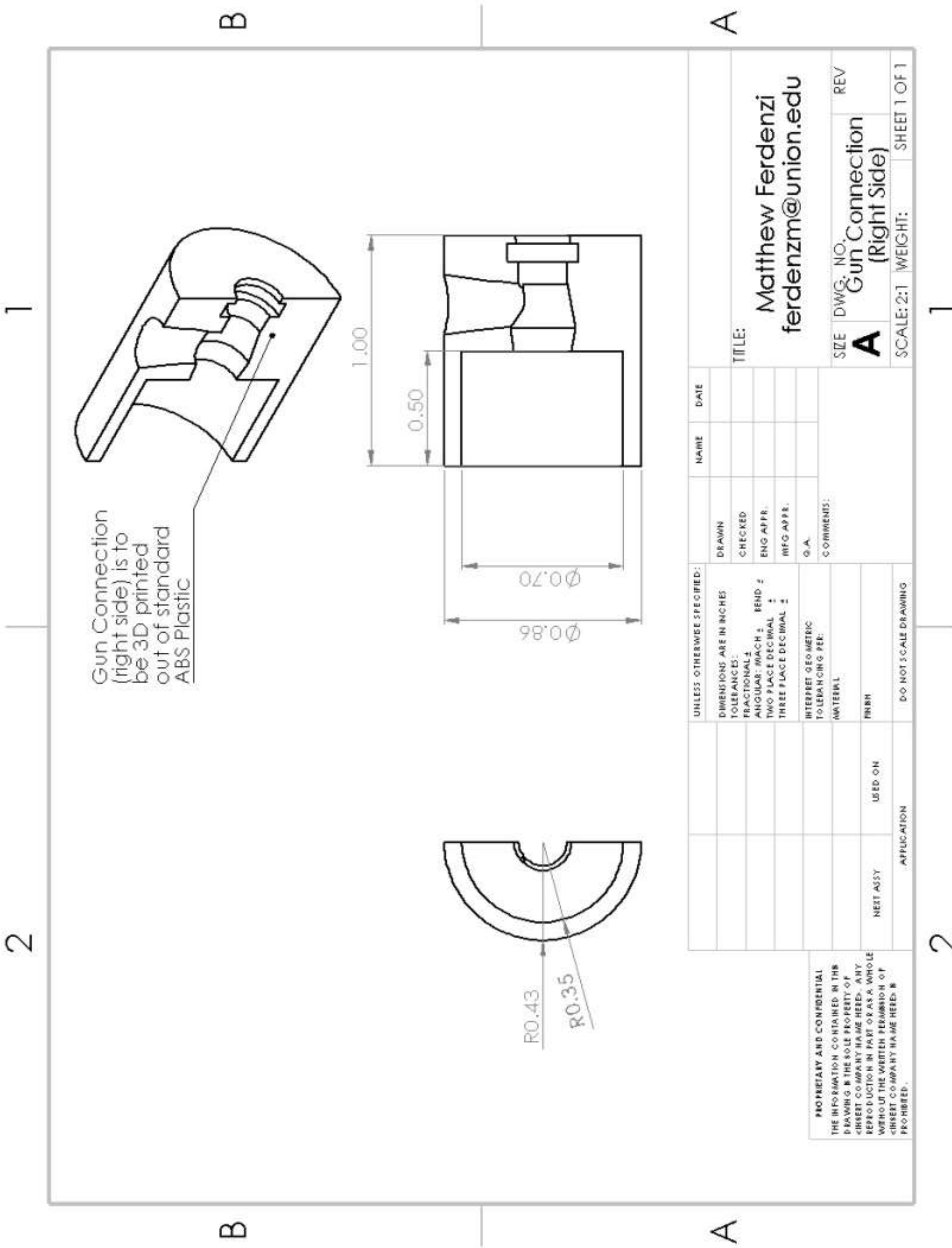

Figure C.7 SolidWorks drawing of the right side of the gun connection.

C.8 Online Appendix

All SolidWorks Drawings and files can be found in the Online Appendix. This appendix is located in the Aerogel Student Research Google Drive, under folder Matt Ferdenzi and then under the folder Online Appendix. The videos referenced in the above report can also be found there. All SEM images taken are provided as well. The link for the Online Appendix is as follows:

https://drive.google.com/drive/folders/1bBeOXUTu2997ju4QeHHmqG4KUaTn0Zin

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