# FINAL REPORT

#### Team 19

#### CNT Reinforced Ceramics 3D Printer



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## ABSTRACT

Currently, there is no 3D printer that prints a polymer that is reinforced with Carbon Nanotubes (CNT). The process of creating this type of material is done by casting, which wastes the expensive CNTs. The importance of being able to produce these CNT reinforced parts by 3D printing saves money since it is done by additive manufacturing, where no material goes to waste. CNTs are extremely unique compared to other fibers because of their high mechanical properties and their high thermal and electrical conductivity. These ceramics reinforced with CNT would be applicable in high temperature applications, such as sensors for monitoring turbines. Team 19's sponsor, Dr. Cheryl Xu, has requested the team to retrofit an existing 3D printer that will be capable of extruding a polymer based material mixed with carbon nanotubes and a ultraviolet light curing reagent. Once a part has been 3D printed and cured, the printed part will undergo pyrolysis in order to achieve a ceramic part which is reinforced by the carbon nanotubes (CNTs). To proceed with the project, a decision matrix was used in order to determine key components, such as what type of 3D printer the team would modify, method of extrusion, and method of curing the polymer. Experiments were done to minimize the line width of the polymer mixture, as high resolution was a high priority for the sponsor. Also, experiments were done by trial and error to determine what volume fraction percentage of CNTs in the polymer would create a high enough viscosity so that the polymer would take its form and not disperse when being extruded. The assembly of the modified extruder includes a syringe pump that is driven by a stepper motor, and underneath the syringe needle a platform is mounted for the attachment of UV bulbs used for curing.

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**Dr. Yong Huang** from the University of Florida has provided information taken from his own experiences from 3D printing to the team's efforts. With so many possible methods of extrusion available and with such a unique material to extrude, the issue of how to best extrude the ceramic polymer composite was resolved in a discussion.

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## 1. Introduction

Optimizing manufacturing processes has been and will continue to be the ultimate goal for all industries. These processes have been revolutionized with recent advancements in 3D printing. It is a favored method for additive manufacturing, which is ideal when conserving material, time, and energy is crucial. By extruding a material in thin lines and stacking each line layer by layer into the desired form, virtually anything can be printed into existence. This has the potential of simplifying deeply involved and highly specialized manufacturing methods in fields such as biomedical engineering, military and aerospace, and even food production because it uses only the material required for each product and it cuts out the need for micromachining and other tedious secondary handling.

Designing a specialized 3D printer was Team 19's mission. The basis of this mission was a novel liquid material involved with Dr. Xu's research and interest. This material is a carbon nanotube (CNT)-reinforced ceramic-polymer composite with unique material properties used in highly sensitive sensor applications. The CNTs are what make the material so unique: by combining these tiny tubes with the ceramic, the ceramic material properties - mechanical strength and electroconductivity amongst others - are amplified immensely, making its industrial potential highly desirable. Previous methods of manufacturing this material were time consuming and costly because they implemented subtractive manufacturing methods and machining to obtain the final product. Dr. Xu desired a method that not only simplified this process, but would also lead to optimal quality in the final product. This created a need for a system that implements all the processes into one, which is possible with the help of 3D printing. This report provides detailed information regarding modeling, design, and verification processes for the development of a CNT reinforced ceramics 3D printer. The report outlines every single step necessary for the development of the previously mentioned device, from the background and concept of additive manufacturing and polymer derived ceramics, to the final design that Team 19 is implementing to make possible the extrusion of such novel material.

The following subsections will describe the scope of the project, design requirements, together with the objectives, constraints, and challenges that the team faced while developing the preliminary designs, experiments, and final design.

#### 1.1 Problem Statement

This project intends to modify a commercially available 3D printer in order to print a novel material mixture consisting of a liquid polymer precursor mixed with CNTs. This process involves the modification of an extruder head that works with a fused deposition modeling (FDM) mechanism into an extruder head able to deposit liquid material mixtures.

Additionally, this polymer mixture needs to be cured before extruding the next layer of material. In order to do this, the team will attaching a curing system into the assembly. All this modifications will be further explained along this report.

## 1.2 Design Requirements

Besides the already existing 3D printing, the design of this project includes a modified extrusion head, the implementation of a curing system, and the addition of control and data gathering devices, as requested by the team's sponsor, Dr. Cheryl Xu. The modified extrusion head must be able to print the novel material mixture as accurately as possible, with the best possible resolution. The best possible resolution will be acquired by extruding the smallest possible layer width. The curing system must be able to solidify each of the extruded layers in the least possible time. The curing time will strongly depend on the width of the extruded layer, and the intensity of the curing system.

Finally, the control and data gathering devices include a dedicated computer, a camera, and a thermometer. These were all requested by the team sponsor in order to allow the printer user to record some data from each of the printing works.

## 1.3 Objectives

Listed below are some of the core project objectives, as determined from meetings with stakeholders as well as through collaborative discussions as a team throughout the project:

• Test a variety of extrusion methods to determine the optimal method of extrusion of the polymer precursor to the print stage

• Perform material experiments, and document how the composition and delivery of the material impacts key performance indicators, such as print resolution and curing time

• Determine the feasibility of achieving CNT alignment and outline a method for the alignment of CNTs in solution

• Test different curing methods to determine the optimal method of curing the polymer precursor to cure and solidify the liquid polymer

• Modify the extrusion head of the 3D printer for permitting the best use of the extrusion method to preserve the polymer precursor material characteristics

• Purchase and install ancillary sensors as indicated by the team sponsor

• Apply sound design principles to all modified parts, including the use of standardized design methods and technical tools like Computer Aided Design (CAD) software

• The printer should be able to generate a printed part that is cured and solidified on the print stage, with the maximum size of a part being no smaller than two centimeters in its widest dimension while also consisting of stacked, self-supporting layers

#### 1.4 Constraints

One of the defining parameters of a project is the limitation of available resources. During the progress of the team's project, important financial, informational, temporal, and physical constraints on the ability to meet project objectives were identified, and are listed below:

• The team was given a budget of 5,000 USD with which to acquire all relevant components, materials, and tools required to manufacture the device

• The project was to be assembled and hosted in a lab of the sponsor's choosing

• The print material should consist mainly of the polymer precursor and multi-walled CNTs in solution; modification or replacement of the key material components would only be possible in extreme circumstances and with explicit sponsor approval

#### 1.5 Challenges

The polymer precursor that serves as the basis of the print material is a proprietary compound developed and sold to materials scientists developing applications in high performance fields. Working with such a material will inherently introduce challenges of its own to the project, as published information beyond what is listed on the manufacturer's data sheet is almost non-existent. A substantial amount of knowledge with regards to the handling, mixing, and chemical interactions has had to be obtained through original research and direct questioning of researchers with hands on experience. Additionally, sponsor's requirements slow down the project completion,

because supplementary information needs to be researched. Below is a list of further challenges that the project team has encountered:

• Current lab procedures for handling and mixing the print material are not able to be translated directly to preparing the material for the 3D printer

• Data on the usability of particular syringe and needle sizes to extrude the material is limited

• Mixing and testing facilities in the sponsor's lab are shared with other projects, and some equipment may be in use for extended periods of time

• The university purchasing system by which large expenditures must be routed must negotiate with the non-traditional vendors required by the project, introducing additional lead time

• Due to sponsor unavailability in the first five weeks of the project, multiple scope changes were enforced on the project as information became available

## 2. Background and Literature Review

No ideas are created in a vacuum, and the work the project team has done to develop its 3D printer is no exception. When beginning the project, the team had only general knowledge of many concepts that would be important to the development of the prototype. Online sources, journal articles, and interviews with experts were crucial to fulfill the knowledge gap, and what follows is a summary of that background information, as well as a review of the critical literature.

#### 2.1 Additive Manufacturing

Additive manufacturing builds objects by adding the material layer by layer, constructing an object that can be too intricate for subtractive manufacturing. First the 3D model of the object needs to be made in detail on a Computer Aided Design (CAD). Then this soft document is converted to an STL file (STereoLithography) which means that the CAD design is recreated by triangulated surfaces without color or texture. Then the STL model is checked for any errors for open objects and unnecessary faces or edges on the model. Once that is done a slicer software converts the STL model to be represented as layers and imbeds instructions in form of G-code which communicates between the computer and the machine to operate. In 3D printer's case, the nozzle and the print bed starts heating up depending on what material will be used and the extrusion head automatically goes to the home location it is assigned. When appropriate temperature is reached, printer starts extruding to the designated locations from the nozzle to the printer bed layer after layer. The object is created by many successive layers and G-code lines. After all lines end and there are no more layers to print the nozzle and the print bed goes into the cool down mode. Once the temperature has sufficiently cooled to somewhere between room temperature and twice the room temperature the object can be taken out by a spatula ready to use in most cases.

3D printing technologies encompasses a wide variety of materials. It started out with thermoplastics but as the technology improved using metals and earthenware became common. Recently there have been experiments on food, wood and bio printing widely and the portfolio of the materials used is ever expanding. This has been achieved many years ago during the 1950s by inkjet printing. Inkjet printing was simply the deposition of the ink suspended in a cartridge onto the designated print stage through the nozzles on the cartridge. The springing of 3D printing came

from stereo lithography which would use a beam of laser to solidify the specified parts of the liquid resin sitting in a vat to achieve a desired shape.

Fused Deposition Modeling (FDM) technology gives more insight about the actual physical portrayal of the material being stacked on top of each other depending on the G-code that was mentioned above. FDM is more specifically referred for molten plastic and 3D printing, but the main principles of FDM where the nozzle is outputting the materials where the material is extruded directly on top of a table that is controlled manually or via computer. Based on the literature, if a material can be extruded through a funnel like nozzle and have control over when to stop the flow it can be printed. FDM technology was patented but when the patent expired, 3D printer enthusiasts created an open source community for both codes of the software and the blueprints of the hardware. This helped the technology to improve at an even faster speed due to the idea being open to many different minds all over the globe who want to solve different problems.

The reason why additive manufacturing is being considered is, it does not produce any waste of material; it prints the specified shape without further need of machining. Currently, there is not a publicly known additive manufacturing technology able to print polymer derived ceramic that is reinforced with CNTs, thus Team 19 will be modifying an existing 3D printer that follows the fused deposition modeling (FDM) principle, into a 3D printer able to follow a liquid deposition modeling (LDM) technique. In order to do that adjustment, the team will have to modify the extrusion head together with the printer profile, and add a curing system to solidify each layer of material before extruding the next.

The following sections explain the designs selected for this adjustment, and the assembly process required for the completion of the transformation. In addition, a reliability analysis, and an economic analysis were develop to determine how will the reinforced ceramics 3D printer perform, and how commercially competitive will it be in terms of costs.

#### 2.2 Polymer Ceramics

Conventional ceramics has been present in the engineering world for many years, and they have been used for different purposes due to their numerous advantages including heat resistance, and relatively cheap production cost. However, it also has a relatively low modulus of elasticity as well as a low tensile strength, compared to other types of materials such as metals, and plastics. [1] Ceramic materials are widely used for their properties such as thermal and electrical insulation,

high melting temperature and chemical resistance to name a few. However, pure ceramic materials are have some major disadvantages such as brittle behavior, low fracture toughness, and namely being. This is where the need for improving the ceramic materials come in. Polymer ceramics (preceramic polymers) decrease the disadvantages seen with pure ceramics a substantial amount. This is done by the addition of fibers that connect the cracks on the ceramic like a bridge and reinforce the matrix. Addition of the fibers bridge the cracks that form with normal brittle behavior, polymer ceramic deforms plastically before fracture. Thus toughness improvement is achieved.

Another big improvement from ceramics to polymer ceramics is modified porosity with the addition of fibers. This increases thermal insulation behavior and allows the user to decrease porosity to increase thermal conductivity or increase porosity to increase thermal insulation. Normal ceramics tend to crack or even fracture when subjected to local temperature changes. Added fibers hold the material together much like the mechanical property change even with severe local cracking. Polymer ceramics are also very stable under corrosive environments. This allows ceramics to be more reliable compared to metals under extreme conditions.

Polymer derived ceramics are known to be corrosive, abrasive, oxidative, and creep resistant. Additionally, when mixed with CNTs, their thermal and electrical properties are highly increased as well as its mechanical properties, which can be compared to those of graphite or Al<sub>2</sub>O<sub>3</sub> [2]. The problem with this kind of improved material is that the current production process, produces too much waste, which turns it into an unfeasible manufacturing material due to the high expenses that the waste of material produces. Therefore, more viable manufacturing technologies are being researched for the production of this type of material. Additive manufacturing is one of those production methods.

Application of these improved polymer ceramics are increasing rapidly due to their reliability under extreme conditions such as high temperatures, heavily corrosive and rough surroundings that could lead to wearing. [3]This leads to the use of polymer ceramics in energy generation that requires very high temperatures, aerospace engineering for turbine blades, aerospace engineering for heat shielding on space rockets, support elements for extreme wearing conditions in biotechnology products, and in electrical engineering for micro and nanoelectronics which would further decrease the size of electronics. Many improvements will be possible in energy, defense, information technology, transportation and micro and nanomechanical and electrical systems. [4]

#### 2.3 Carbon Nanotubes

Carbon nanotubes (CNTs) are hollow cylindrical tubes of carbon that whose diameters range from 1 to 50 nanometers wide [5]. CNTs are categorized based on their structure as either singlewalled carbon nanotubes or multi-walled carbon nanotubes which "are a collection of nested tubes of continuously increasing diameters." [6]

This material has the potential to improve the performance of various technologies due to its superior material properties. Currently, CNTs are used primarily for structural reinforcement [6]. For example, CNTs have been added to polymer composites to enhance the stiffness in tennis rackets & bicycle frames. Thus, it can be seen that it is advantageous to include CNTs within a material. CNTs are highly anisotropic in nature due to their large aspect ratio. To take advantage of their anisotropic structure, CNTs should be aligned in the polymer matrix, which improves the polymer's properties in the direction of alignment. By aligning CNT in the polymer matrix, the strength, stiffness, electrical & thermal properties of the composite can be better controlled as compared with randomly oriented CNT in a polymer matrix [7].

An electromagnetic field can be used to induce alignment of the CNTs within the polymer matrix because CNTs are polarizable particles. There are certain controllable parameters for influencing the degree of CNT alignment with electromagnetic field generation, such as the electromagnetic field strength, application time and the frequency of the applied AC field. Experimental research has suggested that there is a strong correlation between an increase in each individual parameter and the alignment of the CNTs. Additionally, AC fields have shown to be better at inducing alignment within CNTs than DC fields as they tend to lead to more homogenous networks of CNTs in the polymer [7].

## 3. Concept Generation

The concepts that were used throughout the project went through many changes as the team listened and implemented the advice of the faculty. This led to the project going through theoretical and physical evolution throughout the project, while providing work for the goals and the objectives the team was tasked with. The driving force behind all the evolutions were the information and inputs coming from the experienced faculty and staff members of the college and the literature reviews of additive manufacturing, polymer ceramics, and CNTs.

#### 3.1 Initial Design Concept

Initially the project was to attend the American Society of Mechanical Engineers IAM3D Challenge which was an opportunity to re-engineer existing products or create new designs that minimize energy consumption and/or improve energy efficiency. The team was thinking of creating a 3D printer to operate in a more sustainable way. Dr. Xu agreed to sponsor the project if the team agreed to use the novel material she wanted to work with, so the team stopped working towards the challenge, switched the scope and picked up the project that was sponsored.

Next the team was faced with the question of whether to keep the idea of creating a 3D printer or to retrofit a commercially existing one. Dr. Shih advised the team that in order to focus on the material properties as best as possible, the team should retrofit a 3D printer. The project team took that advice and the scope was changed once again to retrofitting a 3D printer that can print with the polymer ceramic material infused with CNTs and align CNTs during this process. In the later deliverables Dr. Xu decided not to pursue the CNT alignment at this stage due to time constraints and it would be sufficient for the team to output a 3D printed part with the polymer ceramic infused with CNTs.

## 3.2 Critical to Quality Factors

During the definition of the project, the team worked with the sponsor to identify the aspects of the final product that would most impact the final quality of the product. These Critical to Quality (CTQ) parameters would be in the forefront of all the future design decisions and would assist the team in evaluating the multiple design concepts that were to follow. After initial interviews with the sponsor, the team conducted an in depth investigation of the project CTQs by means of a detailed questionnaire. The questionnaire listed several aspects of product design such as weight, portability, concentration of carbon nanotubes and gave the responder the ability to indicate their relative importance on a five point scale, ranging from 'Totally Unimportant' to 'Extremely Important', and indicating whether a maximization or minimization of that factor would generate the most positive result. For example, the team's interview indicated that Dr. Xu highly rated the importance of a large percentage of CNT in the final composition, and that the percentage should be maximized to the fullest possible extent. Further CTQ items are listed in Table 3-1:

Table 3-1 Selected results from sponsor questionnaire

Parameter	Importance	Direction of improvement
CNT concentration (% by volume)	(5) Extremely Important	Maximize
Curing time	(5) Extremely Important	Minimize
Print resolution	(4) Very Important	Minimize
General appearance and build quality	(4) Very Important	Maximize
Retention of FDM printing capability	(3) Somewhat Important	N/A
Fume control	(3) Somewhat Important	Maximize
Volume shrinkage on pyrolysis	(3) Somewhat Important	Minimize

#### 3.3 Decision Making Tools

Several tools from product design and process quality were used for guidance on the general direction of the project, as well as guiding specific component level decisions for providing key functionality.

#### 3.3.1 Decision Matrix

A decision matrix is used to compare the relative ability of multiple design alternatives to address project requirements. The matrix lists the alternatives on individual rows, and the functional requirements in columns. Each alternative is assigned a numerical value, typically a positive integer range such as zero to five, to represent how well it addresses the functional requirement with higher numbers typically indicating better performance. Additionally the functional requirements may be weighted to indicate relative importance. By assigning weights as a decimal value from zero to unity, the concept of the weighted average can be applied to each of the alternative-requirement pairs, and the result presented in a final column. This weighted average indicates the ability of that design alternative to meet the functional requirements of the project, given the relative importance of each requirement. Thus, by comparing the averages of the design alternatives, they may be ranked by overall utility.

#### 3.3.2 House of Quality

The house of quality, also commonly referred to as a Quality Function Deployment (QFD) matrix is a powerful tool for allocating project resources in order to maximize benefit with regards to CTQ factors. A QFD diagram lists the quality demands of the customer on the central rows of the matrix and the abilities or functions the product or service provider can supply in the columns. The interactions of demands and capabilities will at that point closely resemble a decision matrix. The value added of the QFD diagram is that the interactions of the quality demands and of the process capability are also illustrated to highlight synergistic or contradictory effects, and the relative importance and difficulty of implementation can be calculated. Use of the QFD allows the team to translate the qualitative opinions of the sponsor in quantitative data to use in making technical engineering decisions.

#### 3.4 Evolution of Design

Sections below are showing the evolution of separate parts of the design determined by the decision making tools above, specifically how material curing can be achieved, appropriate extrusion head for the project and the 3D printer that would be retrofitted.

#### 3.4.1 Curing Method Evolution

The team studied two different options that could be used for the curing process of the printing material. These choices include Ultraviolet (UV) light curing, and heat induced curing. Heat curing is divided into two different areas, which are convectional heating, and laser. On the other hand, the UV curing method can be divided into Light Emitting Diodes (LEDs), and UV lamp, among others. These can be seen on Figure 3-1where the curing methods are decomposed into their varieties.



Figure 3-1 Various methods of curing

Additionally, a decision matrix was employed in order to compare the most relevant parameters of each of the methods to determine what kind of curing process the team should implement. By doing this, the team ensures that the final decision will be the best possible choice. This decision matrix is shown in Table 3-2.

	Cure Time	Placement Flexibility	Availability	Area of Effect	Envelope Size	Safety	Total
Weight	0.25	0.1	0.1	0.2	0.2	0.15	1
UV LED	3.5	5	4	3	5	4	3.375
UV Lamp	3.5	2	3	3.5	3	5	2.675
Laser	4	4	3	1	4	2	2.7
Heat	3	3	3.5	4	2.5	3	2.7

 Table 3-2: Decision Matrix for Curing Selection

The curing time factor will definitely be an important consideration for the team's project. At this moment, the team does not have clear how long will it take to cure this kind of printing material, however, the team is intending to reach a curing time similar of that of already existing 3D printers.

The placement of the curing systems is something else that the team had to consider at the time of deciding which method would be better for the 3D printer chosen, and the printing material that will be utilized. As it is visible on Table 1, the UV lamp scored poorly on the placement flexibility parameter, because it is bulkier compared to all other alternatives; so that would be an example to explain the meaning of placement flexibility.

Area of effect is another important factor, since it was decided that a margin of error must be given for the treated area in order to be more successful with preferably no missed spots. If the area of effect is small there can be errors and all the spots may not be treated the same. This led to laser scoring very poor on this criterion as the laser beams are much more focused compared to the other possible selections. Envelope size was also another important criterion since it would be more feasible to deal with small components and again give the user the flexibility to place the curing system where the team thinks is better. UV LED scored very well on this criterion since its incorporation would be consisting of an array of LEDs each 3 mm in diameter. Lastly, safety is always a priority in any lab setting, because this product will be in the lab.

In addition, Team 19 experimented with heat using the polymer composite without the CNTs. A pipette and a hot plate were used for this purpose. Results yielded that the best time achieved with the hot plate was around 80 seconds (See Figure 6). Even without a set parameter for curing time, a cursory knowledge of 3D printing times suggests that this value is not desirable. This caused the team to abandon using heat as the curing method.

The team tried to run experiments with a laser Team 19 had the chance to borrow from our advisor Dr. Guo, but there were several complications. Team 19 was told that the outlets at the college were not similar or adequate compared to the machinery coming from the NHMFL. Safety procedures in between buildings would also be different; Team 19 would need to be trained specifically for operating the laser equipment as well.

Due to mobility and the theoretical effectiveness of the UV LEDs Team 19 decided to proceed with the UV LED alternative for the curing method.

#### 3.4.2 Extrusion Head Evolution

Several alternatives were taken into consideration at the time of deciding which extrusion mechanism would be more favorable for the project purposes. Based on a lot of research, and expert's opinions, team 19 ranked the different options by using a decision matrix shown on Table 3-3 Decision Matrix for extrusion method.

	Variable Flow	Fluid Viscosity	Material Capacity	Min Shot Size	Temperature Resistance	Cleanability	Total
Weights	0.15	0.25	0.2	0.2	0.1	0.1	1
Syringe	5	4	4	5	5	5	4.55
Nozzle	4	5	5	4	5	3	4.45
Pipette	3	3	2	1	3	4	2.5
Cartridge	4	1	4	3	1	2	2.55

Table 3-3 Decision Matrix for extrusion method

Having a variable flow and being able to control input flow is the criteria where Team 19 had some trouble with during the ongoing testing phase. Being precise and having an accurate way of outputting the same amount of material is important for an extrusion procedure. Viscosity of the fluid is the most important factor to determine what extruder head to use, because the polymer composite will need to preserve its properties, and this viscosity is not going to be similar to melted ABS most printers operate with. Cartridge gets the lowest mark due to its possibility to clog due to the micro nozzles, which use heat to activate the fluid inside it. Material capacity is important since having to refill the materials before the printer finishes printing will be counterproductive. Minimum shot size was another difficulty Team 19 discovered during the testing. Using the pipette, the droplets created were too big, which Team 19 theoretically knows will affect the curing time significantly. With the pipette, Team 19 had some control but not enough to mandate how small the droplet size could be.

Temperature resistance was added to the matrix in case Team 19 decided to proceed with heat for a curing method. Finally, cleanability is on the decision matrix since the shelf life for our polymer composite is around 3-5 days and in case of residue, the extrusion mechanism must be easy to be cleaned.

With these criteria and possible selections in mind, Team 19 will be moving forward with the syringe and needle, or nozzle setups. Team 19 will be further experimenting with these methods via testing in order to fully decide what method would be better to implement.

#### 3.4.3 Printer Selection Evolution

After the project scope changed, the team came together and decided what 3D printer should be purchased. Team 19 was initially looking into a generic hobby printer with a lower price range, nevertheless, the team's sponsor Dr. Xu increased the preliminary budget and informed that it would be better to have a high quality printer that will be more durable to the upcoming modifications. All members of the team researched different 3D printers and Team 19 voted within the team to construct a decision matrix chart, which can be seen in Table 3.

	Sponsor Preference	Open Source System	Availability & Lead Time	Value Added Features	Fit and Finish	Extruder Head Clearance	Total
Weight	0.1	0.15	0.15	0.10	0.20	.30	1
Lulzbot KitTAZ	5	5	4	2	5	5	4.55
Aurora	4	4	4	4	5	3	3.9
Mini Kossel	2	5	5	2	2	3	3.2
Maker Gear	3	3	4	4	3	5	3.85

Table 3-4: Decision matrix for 3D printer selection

Open source hardware and software is also listed as a criterion since Team 19 will be taking it apart to attach the custom extruder head to fit the project needs. Availability and lead-time was an important factor, because the team did not want to wait any longer to get to the 3D printer. The value added features parameter is listed to point out different features that could be needed in the future. The fit and finish criterion refers to the exterior appearance of the 3D printer. One of the sponsor's requests was to have a professional and nice look on the printer, as a result, that point was taken into consideration.

Extruder head clearance is considered the most important criterion since Team 19 will be retrofitting the extruder head and there should be enough free space to permit any design Team 19 may go forward with. After revising all these parameters, and all of the available options, the team

decided that the best choice was purchasing the Lulzbot KitTAZ. Refer to Table 3 to see final results obtained.

After getting clearance from purchasing, the department was ready to order KitTAZ but Team 19 and the sponsor were contacted by the department to indicate that the price of both KitTAZ and TAZ was decreased due to Cyber Monday sales. Therefore, Team 19 decided to purchase the TAZ 4, which is an advanced model of the KitTAZ. The TAZ 4 was not considered at the beginning due to the high price it has, however, when the price decreased there were no more doubts about this 3D printer. Refer to Appendix A to see the TAZ 4 specifications.

#### 3.4.4 Carbon Nanotube Alignment Evolution

The team reviewed background research on electric field induced CNT alignment to gain a better understanding of the fundamentals of this application. This method take advantage of the polarizability of CNTs. A dimensionless parameter,  $\lambda$ , is used to describe the between the energy generated by an electric field and thermal randomization i.e. Brownian motion within the CNTs. This parameter,  $\lambda$ , is defined as

$$\lambda = \frac{\pi \varepsilon_0 \varepsilon_1 a^3 (\beta E)^2}{k_B T} \tag{1}$$

"where  $\varepsilon_0$  is the permittivity of free space (8.854 x 10-12 F/m),  $\varepsilon_1$  is the relative dielectric constant of the liquid resin (about 4.9 at 20Hz), a is the radius of the inclusion, E is the applied electric field,  $k_B$  is Boltzmann's constant (1.38 x 10-23JK-1), and T is the absolute temperature" [7]. Experimental research has shown that there is a proportional relationship between an increase in and induced alignment within the CNTs structure. Therefore it is imperative to maximize the energy generated by the electric field when attempting to align CNTs. Noticeable alignment of the CNTs was observed at  $16 \frac{kV}{m}$  and plateaued at  $43.5 \frac{kV}{m}$  [7].

For this project, the team reviewed the feasibility of using a dispersion and alignment technique similar to one that was conducted by the researchers at the High Performance Materials Institute (HPMI), see Figure 3-2. This method consists of a syringe pump that is used to extrude a mixture containing CNTs out of a small needle. A positive charge is applied to the syringe via an alligator clip attached to the needle. The CNTs within the mixture become aligned after contacting a negatively charged print base. After discussing this technique with Dr. Richard Liang, the principal researcher who developed this method, it was determined that this method would not be suitable

for this project. He mentioned that this method produced a relatively low degree of alignment in comparison to mechanical stretching.



Figure 3-2 A picture and schematic of the electric field CNT dispersion system.

Magnetic fields induce CNT alignment in a similar fashion to electric fields. The condition required for alignment is that the energy generated by anisotropic magnetic susceptibility must be greater than the thermal energy of the CNTs,  $k_BT$ . The energy generated by anisotropic magnetic susceptibility is defined below as

$$\Delta E = \frac{\Delta \chi V B^2}{2\mu_0} \tag{2}$$

"[W]here  $\Delta \chi$  is the anisotropy of the magnetic susceptibility,  $\mu_0$  is the permeability in vacuum, B is the applied magnetic field and V is the volume of each particle" [8]. Assuming that the volume

of the CNTs are held constant, it is important to maximize the magnetic field strength and minimize the ambient temperature to increase the degree of CNT alignment. Figure 3-3 is a graph that plots the ratio of the magnetization that is perpendicular and parallel to the average orientation of the nanotubes vs. relationship temperature. А linear between temperature and the magnetization ratio was observed at temperatures greater than 100K, while below 100K there was a great deal fluctuation. Therefore, this graph suggests that the optimal temperature for alignment is above 100 Kelvin or approximately





~173°C. However, it should be noted that the preferred temperature for solidifying the polymer mixture ranges from 100 - 114°C. This discrepancy presented a major challenge for Team 19.

For this alignment method the team discussed using permanent magnets on either side of the print stage that would align the CNTs within the polymer mixture during curing. However, this alignment method typically requires a strong magnetic fields in excess of 7 Tesla to overcome the low magnetic susceptibility of CNTs. This required magnetic field strength can be decreased by "filling the nanotubes with magnetic nanoparticles" which would increase the magnetic susceptibility, i.e. the degree of magnetization, of the material [9].

## 4. Final Design

The final design mainly consists of a commercially available 3D printer that has both open source software and hardware so Team 19 can change the components, as the team needs to. Main changes to the purchased 3D printer will be the extruder head and the CNTs alignment system. Extruder head will need to accommodate to the characteristics of the polymer composite, which would have a completely different consistency from what the printer is designed to print. The design Team 19 has in mind is focusing on the control of the extrusion of the slurry at a controlled speed and with the finest resolution while the extrusion procedure is unobstructed and can perform well.

The design will utilize the housing and the controls of the existing 3D printer and extrude the polymer composite onto the print stage through the extrusion mechanism. The CNTs alignment unit attached to the printing stage will be working while the print head is printing materials, this way the CNTs are projected to be aligning within the polymer composite before the curing phase starts. The curing process is projected to be flashing in pulse mode to fully transform the polymer composite matrix that is in liquid state to its solid state after printing each layer. This will ensure that the polymer matrix would not be cured to its solid state while it is still above the extrusion head. After the extrusion, alignment and curing for one layer, the method will be repeated until desired shape is formed.

### 4.1 Key Product Components

The printer device that the team has designed is a combination of modified subsystems designed to meet the goals of the sponsor. Because of the use of the novel print material and the challenges inherent in 3D printing, many of these components have either entirely bespoke designs or are repurposed commercially available devices adapted into the printer assembly. Below are detailed descriptions of key printer components.

## 4.2 Syringe Pump

This module consists of a syringe mounted into a plastic housing, with the plunger affixed to a movable slider. By turning a length of threaded rod, the slider can apply pressure to the syringe, depositing the contents onto the print stage. This design incorporates a variable flow rate that is controlled by the speed of the stepper motor. Details on the individual parts of the pump are to follow.

#### 4.2.1 Syringe and needle

Material will be contained in the syringe until it is slowly forced out by the syringe pump. 20 mL is the default syringe volume; however, the bracket was designed to accommodate other sizes as they are desired. A one inch, 400 micron needle attaches to the nozzle of the syringe for precision deposition of the material onto the print bed. This was determined to be the optimal gauge needle for this system after numerous tests involving viscosity, flow rate, and line dimensions.



Figure 4-1 The Syringe pump assembly

#### 4.2.2 Motor and Gear Set

The gear set consists of a 10 tooth pinon gear attached to the shaft of the stepper motor and a 50 tooth gear attached to the threaded rod. This 5:1 gear ratio is used to turn the threaded rod resulting in a translational movement of the syringe's plunger insert. The gears are made from 3D printed ABS polymer, and can be considered self-lubricating.

#### 4.3 TAZ 4 3D Printer

The team and the team sponsor decided to purchase a commercially available 3D printer to be

retrofitted to fit the objective profile required. The TAZ 4, pictured in Figure 4-2, was selected after several rounds of analysis of market offerings. The most important selection criterion for the team was having open source availability for both hardware and software. The TAZ 4 includes a RAMBo (RepRap Arduino-compatible Mother Board) microcontroller to drive the stepper motors. The RAMBo is an open/libre hardware solution that allows the user to modify the firmware using the Arduino development environment. The TAZ is currently running using the Marlin



Figure 4-2 The TAZ 4 printer the team is modifying for the project

firmware package, updated Q4 2014. Information on the proper use and maintenance of the TAZ printer can be found in the user manual published by Lulzbot [10].

#### 4.4 Controller PC

The TAZ 4 is able to be operated independently with the installed Smart LCD controller and display module. However, the team has acquired and installed a desktop computer in order to give finer control over the printer and for use in preparing and organizing model files. The PC is a Dell OptiPlex 2500 with an Intel Core 2 Duo processor, 2GB of RAM, and 755 GB installed hard disk space. The printer is connected to the printer via a USB 2.0 B-type connector, which is standard for Arduino based printer platforms. The PC has Windows 7 Home Premium installed as the operating system, and a plurality of software required for 3D printing activities are developed for Windows as well as Mac OSX and Linux. Software installed by the team is listed in the table below.

Application	Purpose
Repetier Host	Printer controller. Allows the user to place 3D objects on the print platform, adjust printer settings, and manually control the printer
Slic3r	Slicing program. Converts STL file information to G-Code to dictate printer path and steps.
Amcap	Webcam software that allows for creation of time-lapse videos and remote monitoring.
Netfabb Basic	3D model viewer/editor. Repairs STL file meshes for use with the 3D printer.

Table 4-1 Installed Software Used for 3D Printing

## 4.5 UV Lamp Array

The print material as supplied by the manufacturer is a colorless liquid thermosetting polymer precursor. The precursor can be cured to its solid form via ultraviolet radiation in the presence of a suitable reagent called a UV sensitizer. To deliver the UV radiation, the project team has repurposed a commercial nail dryer commonly used to cure resin based nail polish. The core of the product is four 9W UV fluorescent bulbs and the appropriate starters and electronic control components. The back panel features a main power switch, timer selector switch, and start button. To manually cure, the main power switch should be turned "ON", the timer switch set to "OFF", and the start button depressed and released. The timer switch has two other settings, for 120 and

180 seconds of illumination respectively. These settings may be useful for experimental purposes, but are of limited utility in the context of a 3D print job. The bulbs emit UV A radiation centered on a wavelength of 365nm. Additional details of the dryer including manufacturer information can be found in Appendix C, item C2 and a photo of the installed array can be found in Figure 4-3.



Figure 4-3 The installed UV lamp curing array

#### 4.6 Webcam

The project team has incorporated a webcam in order to remotely monitor the 3D printing process and to record images or videos for documentary purposes. The webcam is a Logitech C920 HD camera connected to the PC via a USB 2.0 connection. The camera is a plug and play device under Windows 7, and should not require drivers or other setup by the user. The webcam is attached to the lab countertop via a gooseneck camera mount. Using the mount it is possible to position the camera to tightly focus on the object of attention.

#### 4.7 Infrared Thermometer

Sponsor requirements indicated a means to test and verify part temperature during the curing process. This will be achieved by using an infrared thermometer. The Etekcity Lasergrip 630 thermometer allows the user to get temperature readings without the use of a contact probe. To use the Lasergrip, aim the sensor window at the part to be measured, and pull the trigger. A red laser indicator dot will appear to guide the user on what area is being measured. If the indicator dot does not appear, use of the "MODE" button will cycle between usage modes, including turning the built in back light on and off. Additional buttons will change the temperature display, from holding at last value, to constant updates, and the display units from Fahrenheit to Celsius. Additional technical details, such as measurement range and distance to spot ratio, can be found in Appendix C, item C3.

# 5. Product Assembly

The printer device will be delivered in an assembled state, and thus should not require further assembly unless changes have been made after project completion. This section will list the procedures, tools, and equipment relevant to different assembly operations.

## 5.1 TAZ 4 Printer Assembly

The TAZ 4 is shipped as a semi-assembled unit by the manufacturer which the customer puts together 4 sub-assemblies together and the 3D printer is ready for a print job. The construction materials include 8020 Aluminum square rails, printed ABS components, acrylic corner brackets, and sheet steel electronics enclosures. The primary type of fasteners used are socket head cap screws with metric threads and sockets. The TAZ ships with a small tool set that includes an assortment of hex keys that can be used to adjust and take down the machine.



Figure 5-1 A model of the full 3D printer assembly with significant dimensions labeled

### 5.2 Syringe Pump Assembly

The syringe pump assembly consists of printed ABS parts, stainless sheet steel, injection molded polymer components, and hardened steel smooth and threaded rods. The steel thrust rods are press fit into the receiving holes of the ABS rod guides, and the plastic components are secured to the sheet metal stiffener plate via Phillips drive cap headed self-tapping screws.

## 5.3 Curing Lamp Array Assembly

The curing lamp array consists of four U-type fluorescent bulbs mounted in plastic fixtures attached to a 3D printed ABS bracket that fixtures the lamp array to the extruder carriage. The fasteners used in the assembly of this part are 4-40 round head slotted machine screws and matching nuts. The bulb fixtures have two mounting holes each and the screws pass through holes in the holders and the bracket and are secured with nuts on the side of the bracket opposite the lamps. The bracket holding the lamps is then attached to the carriage using the same type of machine screws, with nuts embedded in the carriage accepting the screws after passing through the bracket. Assembly/disassembly of the array system can be accomplished with a long precision slot head driver and needle nose pliers. The curing lamp wires are connected with spade type wire connectors to allow for service, with wires being routed through holes in the mounting bracket designed for that purpose.

# 6. Operation Instructions

In order to print a model using the ceramic-polymer material, follow these instructions.

- 1) Verify that the syringe is filled with the material and properly fitted into the syringe pump bracket on the 3D printer. The needle should not be touching the printer bed.
- 2) Turn on the PC and 3D printer (using the switch on the power box) for operation.
- 3) Open Repetier-Host on the PC desktop and click "Connect" to connect to the printer.
- 4) Import the model into the program.
  - a) Alter the object placement using "Object Placement" tab as desired. Keep in mind how the object will be printed when doing so.
  - b) If not already sliced, slice the model with Slic3r (using "Slic3r" tab).
- 5) Access "Manual Control" to ascertain all settings are properly set. This includes setting the printer bed temperature to 0° to ensure no heat is introduced to the system. When G-code is ready, send it to the 3D printer by clicking "Send".
- 6) When ready to print, turn on the curing system array with the power supply switch. For added safety measures, UV protective goggles are provided and recommended for use. Once the array is on, click "Start Print". If you will be using the webcam, this would be the time to turn it on. This can be done from the PC desktop.
- 7) From this point the printer will proceed to extrude and cure a 3D model using the material. If this is a large model, then it is highly recommended that the print process is observed and the user is ready to refill the syringe when it gets low. The print job must be paused by clicking "Pause Print". If it becomes necessary to stop the print altogether due to a failure of the print job, click "Kill Print".

In an emergency, cut all power to the printer by unplugging the power strip from the wall. If trying to print using the ABS filament, you must switch the brackets so that the proper extruder is attached. Be sure that the printer is turned off completely, with power unplugged, before doing so to prevent any damage or harm. Instructions for how to use the system with FDM can be found in the manual or online at https://www.lulzbot.com/. No notifications will be given when the print job is complete, nor will any notification be given if a print job goes wrong, whether the layers begin collapsing or the syringe runs out of material, the only way the user will be made aware of an issue is if they observe it themselves. A webcam has been set up with the PC for remote observation access to the printer from within the school so that the print job can be monitored.
# 7. Design of Experiment

The experiments were performed using a programmable pump that would simulate the behavior of the final design. The experimental apparatus for all the tests can be seen in Figure 7-1. There were multiple experiments that were performed and certain procedures that were followed for each different type of test. Before extrusion of the print material could occur, the team had to determine the percentages UV curing powder and nanopowder that was required to mix with the pure polymer. The percentages were found by trial and error. After the slurry was mixed, then extrusion of the print material could be started.



Figure 7-1Test apparatus including the programmable syringe pump

The main goals of the experiments were to achieve the

highest resolution of the print material and the fastest curing time possible. The smallest needle gauge was the driving force for the highest resolution. The thinner the line, the higher the resolution would be. Extrusions were done with various needle gauges of different types of print material. After the first layer was extruded, the team would proceed to curing the print material via UV lamps or UV LEDs. There was a couple of input functions that were kept at a constant. After trial and error, flow rate was found to be constant at  $0.5 \frac{mL}{min}$ . The distance from the end of the needle to the print stage was at a constant height of 3mm. Extrusion droplets and lines were recorded as well as the curing times, and a digital caliper was used for the measuring tool.

## 7.1 Pure Polymer Testing

Team 19 began their experiments by only using the pure polymer to be accustomed to the experimental setup and to understand the polymer ceramic behaviors. The pure polymer the team used is a polymer of very low viscosity; in the range of 50 -200 cps (centipoise) [11]. Relative to other fluids the viscosity of this polymer is similar to blood or olive oil. Initial experiments began with mixing the pure polymer with different percentages of the UV curing reagent. Pure polymer was tested with first because it involved no other variables and the sponsor wanted to see if there was a possible method of decreasing the droplet size and printing this material alone. 4%, 8%, and

16% mass fraction of the curing reagent was mixed in with the pure polymer. As the percentage of the UV curing reagent increase, this decreased the curing time required. Figure 7-2 gives a visual of the low viscosity pure polymer.

## 7.1.1 Results

The first thing that was measured and recorded was the diameter of the droplet size when it was being extruded from the programmable syringe pump setup. For this setup, the needle used for extrusion, 30 gauge needle (156 microns), remained constant. This was the smallest diameter needle that the team was able to find on the internet that could be purchased through the school. For this experiment the flow rate was varied to determine a controllable flow. The height from needle end to print stage was constant at 3mm. From Figure 7-3, it can be seen that the flow rate varies from  $0.1 - 3.0 \frac{mL}{min}$ . Also, the graph shows no real correlation that changing the flow rate would decrease the diameter of the



Figure 7-2 Extrusion test of the pure polymer precursor

droplet size. Since there was no correlation to decrease the droplet diameter with manipulating the



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flow rate, the team decided that a slower flow rate would be more appropriate to test with for better control of the material. When the material was extruded, it was not able to hold its form and it dispersed rapidly. The average droplet size was approximately 3mm, which is substantially larger than the needle diameter. Also, due to the low viscosity of the polymer, the polymer would start to leak out of the needle even if there was no load applied to the syringe.

Table 7-1 shows the results of curing the polymer with different percentages of the UV curing reagent. A plethora of droplets were dropped onto the print stage as the UV LED board hovered 3mm above the print stage. The droplets were checked to see if curing occurred about every 4 minutes. As expected, the higher amount of UV curing reagent added the quicker the curing time.

UV LED Curing							
Mixture	avg. diameter of droplet (mm)	# of runs	time (min)	total time elapsed (min)			
		1	4				
		2	3.5				
Polymer mixed w/ 4% 2-2 DP	4	3	4.0	26.50			
		4	3.5				
		5	4.0				
		6	4.0				
		7	3.5				
		8	4.0				
		1	4.0				
		2	5.5				
Polymer mixed w/ 8% 2-2 DP	1	3	4.0	21.5			
		4	4.0				
		5	4.0				
Polymer mixed w/ 16% 2-2 DP	1.75	1	8.0				
		2	4.0	12.0			

Table 7-1 Curing times for lines of different diameters and compositions

# 7.2 Surface Tension Testing

Surface tension testing method and how the idea was generated was detailed below. The team had to get familiar with fluid movement within a small sample. The behavior was somewhat unlike the fluidal mechanical engineering classes due to not having a continuous flow. To understand the aspects better the team reached out to the designated advisors.

## 7.2.1 Print Stage Material

It was suggested by Dr. Wei Guo that if the team could minimize the surface tension the droplets would become more spherical and bead up instead of dispersing. This could be done by minimizing the higher energy bonded molecules to achieve the smallest surface area to volume ratio. Dr. Guo also suggested to see if there was a way to relate the contact angle to the pressure and the velocity of the flow. However, Dr. Sam Taira mention to the team that potentially trying to use different types of hydrophobic print stages may give a smaller diameter.

#### 7.2.2 Results

The team went with the route mentioned by Dr. Taira because it was the quicker method to experiment. The various hydrophobic surfaces the team used included: masking tape, parchment paper, glass with rain-x, and Teflon. Figure 7-4 shows the various surfaces that were tested. The main point of the graph is to shows that the glass with the rain-x would be the most viable option



Figure 7-4 Scatter plot of droplet diameter for different substrates

to minimize the droplet diameter. However, even though glass with rain-x produced the smallest diameter; the diameters were still significantly larger than what would be needed to produce a high resolution print. At this point the team was able to safely remove the idea of just printing with the pure polymer and to move onto the next step, which was adding nanopowders into the mixture.

## 7.3 Carbon Nanotube Testing

This portion was crucial to the project since the addition of CNTs was one of the objectives of the project and it is crucial to understand the behavior of polymer ceramic with CNTs imbedded.

#### 7.3.1 Varying Percentages of Nanopowder

This was the primary nanopowder that the sponsor wanted to mix into the polymer with the UV curing reagent. At this point in the experiment alignment of the CNTs were neglected due to time restraint and materials that would be required to induce a small amount of alignment. The team's main focus was to obtain a print material that was more viscous than the pure polymer and behaved more like the FDM 3D printer, which meant extruding by lines instead of droplet by droplet. The first thing that needed to be determined was the mass fraction percentage that would be needed to give the polymer a paste like viscosity for line extrusion. This was done by trial and error starting and 1% mass fraction and working up until the team decided it was viscous enough. At 2% the team determined that this percentage would be viscous enough for the final print. A theoretical calculation was performed using Poiseuille's Law in order to determine the viscosity of the print material and can be found in Appendix B.

#### 7.3.2 Varying Needle Gauge

Initial testing with the polymer mixed with CNT cause the programmable syringe to stall due to high pressures. The cause of stalling was because the 30 gauge needle was too small in diameter resulting in high pressures that the stepper motor could not handle and also caused elastic deformation to the syringe pump holder. The team decided to put in a purchase order for different sized needle gauges: 18 (838 microns), 20 (603 microns), and 22 (400 microns) gauge needles. The larger diameter gauges all operated with the programmable syringe pump.

#### 7.3.3 Results

The first experiment that needed to be completed was to determine which needle gauge outputted the best resolution. Figure 7-5 shows the experiments that were performed with different needle gauges. The graph is a box and whisker graph where the bottom line is the minimum, the next line up represents the first quartile, the next line up is the median, the next line up is the third quartile, and the top line is the maximum. From the graph, the team has decided to move on with the 400 micron needle for the final design barring any setbacks from the stepper motor or gear ratio. If high pressure causes stall the 603 mircon needles may be used. Also, the different length needles came in the shipment, so the team decided to test with them and see if there was any significant difference. It can be seen that the main difference is the minimum and maximums. The one inch needle have less of an outlier due to no-slip conditions at the walls inside the needle being longer, which would increase the friction.



Figure 7-5 Box and whisker plot for line widths from different guage needles

Figure 7-6 shows a visual of the polymer mixed with CNTs being extruded by line extrusion instead of droplets. Being able to cure the CNTs mixed with the polymer was the last step of the experiment. When curing the CNTs mixed with the polymer, the team decided to use UV bulbs instead of the UV LED arrays. The UV LEDs was a complicated setup and not providing enough power to decrease the curing time. The chart in Figure 7-6 compares different curing methods as well different power outputs. The first section of the graph refers to the curing of the pure polymer via LEDs, the second section refers to a single 9 Watt bulb used to cure lines of about 1 mm in width, and the last section has a power output of 36 Watts and cures a 0.81 mm width line in 2.5 minutes. This suggests that the higher the power output the faster curing may be achieved.



Figure 7-6 Syringe loaded with CNT mixture



#### **Trial Amount vs Curing Time**

Figure 7-7 Line plot illstrating curing time under different scenarios

# 7.4 Silicon Carbide Testing

Silicon carbide powder was suggested to be tested by our sponsor and advisor Dr. Xu after the team expressed concern for using highly priced CNT powder liberally. The team conducted tests with this powder but the properties of the matrix made by silicon carbide did not reflect the CNT matrix behavior. Thus the experiments with this powder was concluded.

#### 7.4.1 Varying Percentages of Nanopowder

Silicon carbide was suggested as a secondary nanopowder to experiment with by the sponsor. The reason for experimenting with silicon carbide is the fact that it is a cheaper nanopowder than CNTs. The main reason why CNTs have not been implemented in 3D printing in the manufacturing industries is because of the high cost of CNTs. The team did another trial and error to find a mass fraction percentage of silicon carbide that would behave like the 2% mass fraction CNTs mixed with the polymer. The team went as high as 17% mass fraction of silicon carbide. However, since the team found the 2% CNT mixture first, the silicon carbide was of less importance and put on hold.

#### 7.4.2 Results

When testing with the 17% silicon carbide mixture, the experiments were nearly identical to the CNT mixture experiments. The first thing the team looked for was the best fit needle gauge. Figure 7-8shows that the 400 micron needles would be the best choice for resolution. The graph depicts a box and whisker as explained before, except the 400 micron box plot may look strange because there are only two regions. The first box plot has the same median as the third quartile, so that is why there are only two regions instead of three. From the experiment it is clear that either lengths of the 400 micron needle could be used. After these sets of experiments the team stopped testing with the silicon carbide because the mixture of CNTs and the polymer were found to be adequate. If further experiments were to continue with the silicon carbide, the percentage of silicon carbide would need to be increased. From experimenting, the silicon carbide would tend to leak out of the needle when the print has stopped or paused due to gravity and its viscosity. A certain percentage could be calculated using the MATLAB code in Appendix B to match up the viscosity of the silicon carbide mixture to the CNT mixture.



Figure 7-8 Box and whisker plot showing line width generated from different gauge needles

# 8. Considerations for Environment, Safety, and Ethics

This section presents environmental concerns, safety information linked with the chemical materials involved in the 3D printing process and listing possible hazards that the project may contain.

## 8.1 Environmental Concerns

One of the top priorities of the project team was to design a product as environmentally friendly as possible. Thus, at the time of selecting which components would be forming the final product, this was an important parameter to take into consideration. Additive manufacturing itself represents one of the greenest production processes compared conventional manufacturing processes. As a result, the greatest concerns fall back into the printing material mixture components. Many existing 3D printers deal with thermoplastics, which can be re-melted and shaped again as a recyclable material. However, the material is not a plastic. The material is a polymer derived ceramic combined with CNT powder; thus the disposal of this material may not be environmentally safe. The following subsections will explain in depth the disposal and exposure risks for the CNTs and for the polymer precursor.

#### 8.1.1 CNT Disposal and Exposure Risks

According to the National Institute of Occupational Safety and Health (NIOSH), there is no evidence of adverse health effects in workers producing or using CNTs; however, there are studies performed on animals that indicate possible health adversities for people exposed to CNTs. Some of these exposure risks include pulmonary inflammation, pulmonary fibrosis, and cancer. Since there is no certainty regarding the risks of being exposed to CNTs, it is recommended to take a precautionary attitude.

First of all, it is important to wear gloves at the time of preparing the polymer mixture to be extruded during the printing process. In addition, safety glasses are recommended to avoid the contact of any material with the user's eyes.

For disposal matters, it is recommended to wrap the CNT waste in sealed polythene bags and gathers it in a well-labeled waste container. It is not recommended to dispose CNTs in the sink, or in conventional wastebaskets [12].

#### 8.1.2 Precursor Disposal and Exposure Risks

Studies show that there are not many risks related to the exposure of this polymer precursor. However, in order to avoid accidents it is important to store the material in a closed container whenever it is not being used. It is also important to avoid its contact with the eyes, skin, and clothing, as well as the repeated exposure.

According to the Occupational Safety and Health Administration, this material is not carcinogen, and not many studies had been conducted with this type of material. However, studies performed on animals agree that the repeated exposure or contact with this kind of polymer might cause skin irritation.

For disposal matters, it is not recommended to combine this material with aqueous wastes [13]. Since this material is considered combustible waste material, incineration of the waste is the recommended disposal method.

## 8.2 Ethics

The design that Team 19 is using for the modified components is completely new, therefore there is not a copyright infringement regarding components design. Additionally, the team is taking all the safety and environmentally hazardous issues that the printing process might cause into consideration in order to avoid any potential accidents or unhealthy experiences to the users.

Polymer derived ceramics have shown to have improved properties in terms of strength, thermal conductivity, and temperature stability even without the addition of CNTs. As a result, there are many applications in which the implementation of this type of material would be extremely beneficial. Some of the advantages that this type of material could offer in the long run include technological advancements in the research field, and safer working equipment for applications such as aviation, and heat demanding technologies, among others. The implementation of this relatively new material into the additive manufacturing world will be highly beneficial for the development of new technologies, and improvement of several applications.

## 8.3 Safety Information

Despite the best efforts of any designer, no equipment can be made entirely safe. Every time an operator uses a piece of equipment there is the chance of a health or safety impacting event. Part of any risk mitigation strategy is to ensure that operators understand the potential risks involved in the operation of a piece of equipment, so that they can protect and educate themselves. Team 19 has identified some of the risk factors that may be present while operating the device which are available below.

#### 8.3.1 3D Printing Hazards

In its default configuration, any 3D printer poses a number of safety hazards to the user. The use of heated, motorized, and electrically active components means that caution should be exercised whenever using, modifying, or repairing the TAZ printer. General safety warnings from the manufacturer of the TAZ 4 printer are reprinted in Appendix A. Even with the current configuration with no heat, no modifications performed by the project team should be assumed to reduce or eliminate any of the risks posed by use of the printer.

#### 8.3.2 Carbon Nanotube Hazards

Carbon Nanotubes (CNT) are microscopic hollow structures comprised mostly of carbon that exist at the scale of a nanometer, or one billionth of a meter. The exact hazards posed by exposure to carbon nanotubes is currently under experimentation, with animal studies indicating "that as a precautionary default: all biopersistent CNTs, or aggregates of CNTs, of pathogenic fibre dimensions could be considered as presenting a potential fibrogenic and mesothelioma hazard unless demonstrated otherwise by appropriate tests..." [14]. Thus the proper handling and use of nanomaterials should be a priority in the operation of the prototype device. As no exact type of CNT is indicated for exclusive use with the printer, users should be aware of the type of nanomaterial being used with the polymer precursor, as the risks inherent to each type will vary with the characteristics of the material. Points of consideration include: whether the CNT are single or multi-walled, the quantity to be used, the aspect ratio of the CNT, how tightly bound the CNT are to each other, and have they been functionalized or treated with any substance that can affect their health risk [15]. Make sure to dispose of the chemical into the appropriate waste jar.

#### 8.3.3 Ultraviolet Radiation Hazards

The printer device uses lamps that emit electromagnetic radiation in the ultraviolet (UV) spectrum. The lamps emissions are tuned to emit primarily light with a wavelength of 365nm. This wavelength is within the UVA band, which is considered to be among the least harmful types of UV radiation. However, precautions should be taken in the presence of any UV radiation source. Long-sleeved clothing should be worn, as well as eye protection in order to minimize exposure. Polycarbonate lenses and face shields will block 99% of UV radiation, and surface treatments and films can increase protection further. The American Conference of Governmental Industrial Hygienists has set Threshold Limit Values (TLV) for tolerable repeated exposure levels to UV radiation. For energy in the UVA band, the TLV is approximately  $1000 \frac{mJ}{cm^2}$  [16]. The UV lamps installed on the printer dissipate a nominal 36W of power; thus indicating that if one square centimeter of skin is exposed, the TLV could be exceeded in 0.028 seconds. This does not take into consideration the insulating effects of air, which could extend the safe exposure time limit.

#### 8.3.4 Polysilazane

The main composition of the slurry is the polymer, and the chemical identity of the polymer is called Polysilazane. There are safety precautions that have to be considered when using this polymer due to the chemicals involved. Corrosiveness, flammability, and severe eye and skin irritation are the potential health effects due to overexposure in the workplace. Contact, inhaling, or ingestion of the chemical are the primary routes of exposure. If exposed there are first-aid measures that need to be taken to prevent further irritation. If the chemical makes contact with the eyes, immediately flush eyes with water for at least 15 minutes. If the chemical comes in contact with the skin, wash the exposed part of the skin with soap and water immediately. If the chemical is inhaled, remove to fresh air. If breathing becomes difficult use artificial respirator or supply a source of oxygen. If the chemical is accidently swallowed, seek medical treatment. Once exposed and irritation persists after initial treatments, seek medical assistance.

Other measures need to be taken into account besides exposure, such as spills and firefighting measures. If a small spill occurs, cover the spill up with vermiculite, perlite, sand, or clay, and sweep it up and dispose appropriately. If a large spill occurs, use a structure to contain the spill and pump into drums for use or disposal. Clean up the remaining area with soap and water. If a fire occurs, extinguish with carbon dioxide or a dry chemical powder. If the container is on fire,

use water to extinguish. To prevent and control exposure or fires from breaking out wear normal laboratory protective clothing. For instance, lab coat, safety glasses, gloves, and a fume mask. Also, working under a fume hood or closed booths is recommended. Have appropriate hygienic practices; wash hands thoroughly after handling the chemical. Make sure to dispose of the chemical into the appropriate waste jar.

#### 8.3.5 UV Curing Reagent

The UV curing reagent that is mixed into the slurry is called 2, 2-Dimethoxy-2phenylacetophenone. There are a couple hazards to be aware of; the chemical may cause an allergic skin reaction, and the chemical is very toxic to aquatic life with long lasting effects. Before experimenting with the chemical take precautionary measures. Wear laboratory clothing, such as gloves, coat, glasses, mask, and work in a fume hood. Avoid inhaling chemical, release on the environment, and exposure to skin. If exposed to skin, wash with soap and water. If the chemical inhaled, seek medical attention. If irritation persists due to exposure seek medical assistance. If a fire occurs: use water spray, alcohol resistant foam, or carbon dioxide.

From the toxicological information, the chemical has been tested on mammals and there are no serious results that leads to harming a person or the mammals for skin and eye exposure. The chemical is toxic to fish and other aquatic invertebrates. Dispose of the material in the appropriate waste container. Do not dump the chemical down a sink. Make sure to dispose of the chemical into the appropriate waste jar.

#### 8.3.6 Nanopowder

The nanopowder that will be mixed in with the slurry is multi-walled carbon nanotubes. From the hazardous materials identification system, the health hazard rating is 2, the flammability is a 0, and the physical hazard is a 0. The ratings range from 0 - 4 where 0 being a non-threating material and 4 being a very hazardous material. The only health hazards from this material is that it may cause eye irritation as well as respiratory irritation. To prevent these health hazards wear laboratory clothes such as gloves, coat and mask and work in a fume hood. When working with the chemical, avoid breathing in the dust and wash hands after handling. If the chemical is inhaled remove to fresh air environment. If the chemical gets in eyes, rinse with water for several of minutes. If irritation still occurs for both cases, seek medical assistance. Make sure to dispose of the chemical into the appropriate waste jar.

#### 8.3.7 UV Curing Information

The polysilazane may be cured by UV radiation if a UV sensitizer is present, and for this experiment there is a UV curing reagent added to the mixture. The liquid-state resin is cured by absorbing the UV radiation when the photopolymers are excited. The activated photopolymers react with the resin and a three-dimensional cross linking occurs and molecular weight is increased. UV curing can be done with LEDs, lamps, or bulbs. For this project, bulbs will be used due to some constraints with the other UV curing methods. For LEDs, there was not enough power output that can be generated; and for lamps, the dimension of lamps were too large to fit in the print area. For the design project, Manual droplets and lines were created by a syringe pump. The manual line width was simulated to match the programmable syringe pump. The smallest width line was 0.6 millimeter and curing of the slurry was estimated to be roughly 8 minutes. This test was done with a single 9W bulb, if intensity of the bulb was increased there would be an increase in curing time.

# 9. Project Management

Below is a breakdown of how project roles were divided among the team members, the approach used in scheduling team, resources the team used for research and procurement, how the budget component was managed throughout the project and the communication methods that the team decided to use during the project development process.

## 9.1 Roles

The roles of each team member were clearly defined at the beginning of the project. These roles were determined based on each member capabilities and knowledge. Six persons formed Team 19; four from the ME department, and two from the IE department. There was a Team Leader, in charge of creating an agenda for each team meeting and assigning tasks for each member if needed. The Lead ME was the official spokesperson for the ME aspect, regarding technical discussions and updates from the ME department accordingly. The other members of the team were in charge of mechatronics, experimental analysis, CAD modeling, and finances, depending on their major strengths.

## 9.2 Scheduling

Most of the project scheduling was handled by using a Gantt chart. The Gantt Chart was divided into five major sections, which were derived from the standard DMAIC Six Sigma paradigm that means Define, Measure, Analyze, Implement, and Control. Since this standard strategy is mostly used for process or product improvements, Team 19 used a modified version of this methodology that applies for the design of a product or process: Define, Measure, Analyze, Design, and Verify.

The Define phase included tasks related to research and clear understanding of the project scope. Some specific tasks included weekly meetings with the team sponsor, study of the different sections of the project, and design of experiments for the following phase of the project. During the Measure phase, the team conducted several experiments and took final decisions regarding components' decision making with the help of different decision matrices. This phase allowed the team to understand the properties of the polymer mixture to be printed, and gain a better notion of the necessary for the completion of the project. On this phase the team began purchasing different

components that would be required for the next phase. Some problems, including finding testing equipment and deciding the optimal components to be ordered, were faced. Nevertheless, the team managed to solve those obstacles and other unexpected situations.

During the second half of the project, the scheduling approach was changed. Phases were no longer used to divide the project tasks. However, the flow of the tasks was basically the same, with little variation. At the beginning of the spring semester, the team performed an in-depth examination of the results obtained from previously performed experiments. Depending on how satisfactory were the results gathered, the team decided to design new experiments, or to perform more trials of the same experiments with varying parameters.

Once most of the analysis was ready, the team began to modify the existing 3D printer. This part of the project took longer than the others, delaying the overall completion of the project due to the uncertain behavior of the component modifications. Therefore, following the critical path became difficult at this point of the project.

#### 9.3 Resources

Team 19 received help in terms of resources from multiple persons; faculty, staff and the facilities. Dr. Cheryl Xu, the sponsor and the main advisor for this project, provided an adequate space in her laboratory facility for the development of experiments, and for safely placing the 3D printer and its secondary components. Additionally, Dr. Wei Guo, who is another team advisor, was an important resource at the beginning of this project due to his knowledge regarding magnetic field alignment.

Other important resources were The High Performance Materials Institute (HPMI), and the machine shop located at FAMU-FSU College of Engineering. Some of the project components were shaped at the machine shop and this was very helpful, because otherwise the team would have to wait in the work queue at the college machine shop which would have been more time consuming. Similarly, the HPMI was a helpful resource towards the end in printing out parts with their 3D printers since the team had the syringe pump component installed and testing. HPMI's research studies and machines also helped the team learn from similar additive manufacturing machines that HPMI contains.

## 9.4 Procurement

For this project, the initial budget was \$5,000.00. The team only spent approximately sixty percent of the total budget. The expenses include the 3D printer purchase, which represents the most expensive element for the development of this project; the curing system components, the extruder head components, temperature sensor, camera, ABS filaments that were used to print several components necessary for the extruder head and curing system assemblies; electrical components, and incidentals.

Appendix F, shows the break down costs of the different constituents of the final product assembly. It is important to mention that some of the components included in this table are not part of the final design; however, they are included on the final cost of this project. Since Team 19 was dealing with a research project, experimentation was an imperative portion for the progress of this project. As a result, some of the components listed in Appendix F were purchased for experimentation purposes; therefore if they did not work as expected, they were not used on the final design. Figure 9-1 shows the percentages of budget allocated for the devices and components purchased.



Figure 9-1 Pie chart of budget allocation divided by category

## 9.5 Communications

For proper team communication, the team decided to meet once a week, and sometimes twice a week depending on the project needs. During these meetings, the team talked about multiple issues including due dates, experimentation progress, and design issues, among others. Additionally, the team tried to set one meeting per week with the sponsor, so she could be updated about the project progress. In addition, all information related to the project was shared with the group by using a Dropbox folder. This information included research papers, reports, CAD files, posters, and presentations. Also, a GroupMe group was created to keep all the team updated at all times, and for more informal information exchange. To communicate with the sponsor, advisors, and other professors, the team used the e-mail resource, which worked as a more formal way of communication. Additionally, to keep the mechanical engineering department informed, biweekly staff meetings were held.

# 10. Recommended Future Work

In reaching the conclusion of the project, Team 19 has discovered multiple aspects and even flaws in the final design of the retrofitted 3D printer in need of further attention. Within the following sections are various discussions of these aspects and suggestions that could be valuable starting points for the team following Team 19.

## 10.1 Carbon Nanotube Alignment

One significant component desired within the retrofitted 3D printer this semester was a carbon nanotube alignment system. By aligning the carbon nanotubes parallel to the printer platform within the ceramic matrix, the material properties of the final model would reach optimum amplification for Dr. Xu's applications.

Through extensive research and discussion, it was realized that this system was unobtainable within the given time period due to the testing and experimentation still necessary to fully understand and design such a system. Multiple methods exist in theory and many of these methods have been tested with varying results; however, consistent results were not successfully obtained in any of these tests, and those tests that had results were only partially successful in aligning the carbon nanotubes properly. It is a project within itself and would have consumed the time needed to test and design the other desired systems.

## 10.2 Material Mixture Refinement

When combining the ceramic-polymer with the reinforcing carbon nanotubes, the material properties, particularly the viscosity, are altered drastically. From thorough testing a feasible ratio of polymer to carbon nanotubes was obtained and further progress was based off of this ratio. In order to extrude the solution from the syringe without leakage, a higher viscosity is desirable. One disadvantage of the higher viscosity was discovered when testing various needle gauges: the volume fraction of carbon nanotubes correlates to the size of the needle in that a higher volume fraction requires a larger gauge in order to prevent clogging in the nozzle. More testing could reveal a more suitable ratio with optimal material properties or would in the very least garner a better understanding of the material. Refining the mixture could very well lead to an alteration in how it is extruded.

## 10.3 Curing Array Improvements

Final curing array testing and designs have concluded with a minimum material curing time of two and a half minutes, with overlapping or oversize structures indicating additional time required to solidify parts. Alterations to z-axis homing were made in order to accommodate this assembly. Observable issues with this system have occurred and have been handled with success as they are discovered, though better solutions are certain to exist.

One issue is the quality of the curing. The syringe extrudes the material onto the printer platform in thin lines. Curing the surface of these lines is quickly accomplished compared to the rest of the material beneath the surface. The UV light has to shine longer on the line in order to penetrate through the surface layer and into the rest of the material, and as the material is stacked layer by layer, the surface area covered by the UV light is decreased, leading to an increased curing time. Sometimes the curing is only partially accomplished and the internal material remains in a transitional solid phase, resulting in a weaker structure that does not maintain its integrity when being removed from the printer platform. In order to counter this issue, a substrate that can be placed in the furnace with the cured model is laid down on top of the printer platform so that it acts as the substrate for the material to be extruded upon. This actually simplifies the removal process so that the final printed model can be directly carried from printer to furnace without any further preparation.

Although a feasible method was determined and implemented to accommodate for the partial curing, it is still not the optimal result Team 19 desires. Another method must exist to better cure the material during the print process on top of further minimizing the curing time. This is still possible and highly desired by Dr. Xu, and improvements to the system will need to be made in order to accomplish this.

#### 10.4 Gear Set and Stepper Motor Modification

A stepper motor drives the gear pair located on the top of the extrusion system, which in turn revolve the threaded rod which forces pressure on the syringe plunger, extruding the material onto the printer platform. During long print jobs, this motor begins to heat up and is at risk of overheating, resulting in the introduction of oscillations to the system and possible failure. The addition of a fan could act as a solution, despite being a perfunctory solution. Multiple alterations to the extrusion system could be made to strengthen it. One could calculate a gear ratio that provide a stronger torque to the threaded rod to provide a stronger force to the syringe, overcoming any opposing pressure originating from the pressure of the material within the syringe.

Another alteration could be swapping the stepper motor for a more powerful one capable of withstanding long periods of use. One could even go back to refining the material mixture as discussed in Section 10.2 in order to ease extrusion further. This aspect would require further discussion and consideration of any other modifications being made to the printer system. It may also have an effect on the following section.

#### 10.5 Continuous Feed Device

In its current incarnation, the printer relies on the preparation and loading of discrete syringes containing all of the material available to print with. As an improvement over this current implementation, a future team could implement a mechanism by which a large reservoir of prepared material is fed continuously to the extruder. This would have the benefit of increasing the maximum size of a printed object and reduce the time spent setting up the printer between jobs. The development of this modification would require a means to ensure that the material maintains homogeneity and does not solidify in the container, as well as providing consistent pressure of a thick semi fluid to the extruder. Additionally, a gauge or other means to visually confirm the contents of the reservoir would be useful to future users.

# 11. Conclusion

3D printing is a phenomenal manufacturing process that allows anything one can design to be brought to existence. When paired with a novel material like the carbon nanotube reinforced ceramic polymer composite, a previously lengthy manufacturing process becomes optimized with a cleaner, more efficient additive manufacturing process and a stronger understanding of the material's properties and potential is more easily obtainable. Team 19's mission was to make this pairing possible, and the team achieved this through a lot of discussion and experimentation.

Numerous diverse methods for extrusion, curing, and carbon nanotube alignment were researched and tested thoroughly, leading to many unexpected results and conclusions. The final retrofitted syringe pump extrusion system was the result of its strength when compared to the other systems considered, as it works best with the high viscosity of the material even offering room for feed rate alteration, and simplifies maintenance. As for curing, the four 9W ultraviolet bulbs have the overall best curing results with the material as of this report and are arranged to cover the greatest surface area of the extruded material possible while evenly directing their combined power to each layer. Success is based on printing and curing a final model representing the capabilities of the 3D printer.

The evolution of Team 19's 3D printer lead to the necessity for adaptation at every turn of the project, and having had a clear definition for the project from the very beginning would have undoubtedly saved time and energy down the road. Nonetheless, the members have taken away an irreplaceable experience from the last two semesters. Room for improvement is possible in all systems involved and suggestions are written in this report in the hopes that future teams will be able to better the system in every way possible.

## References

- [1] RocCera, "Advantages of Ceramics Vs. Steel," [Online]. [Accessed 15 September 2014].
- [2] P. Colombo, G. Mera, R. Riedel and G. Soraru, "Polymer-Derived Ceramics: 40 Years of Research and Innovation in Advanced Ceramics," The American Ceramic Society, Trento, 2010.
- [3] V. A. Lavrenko, Corrosion of High Performance Ceramics, Springer-Verlag, 1992.
- [4] H. Lange, M. Dogigli and M. Bickel, "Ceramic Fasteners for High Temperature Applications," *5th International Conference on Joining Ceramics*, p. 55, 1997.
- [5] "Carbon Nanotubes," nanocyl, 2015. [Online]. Available: http://www.nanocyl.com/jp/CNT-Expertise-Centre/Carbon-Nanotubes. [Accessed 1 4 2015].
- [6] "Carbon Nanotube Synthesis Technology Overview," nanoScience Instruments, 2015.
   [Online]. Available: http://www.nanoscience.com/products/carbon-nanotubesynthesis/technology-overview/. [Accessed 1 4 2015].
- [7] B. Sumanth, C. Park and J. Wilkinson, "Aligned Single Wall Carbon Nanotube Polymer Composites," *Journal of Polymer Science Part B: Polymer Physics*, vol. 44, no. 12, pp. 1751-1762, 2006.
- [8] B. Jang, Y. Sakka and S. Woo, "Alignment of carbon nanotubes by magnetic fields and aqueous dispersion," *Journal of Physics: Conference Series*, vol. 156, no. 1, 2009.
- K. Iakoubovskii, "Techniques of aligning carbon nanotubes," *Central European Journal of Physics*, vol. 7, no. 4, pp. 645-653, 2009.
- [10] Aleph Objects, "TAZ 4 User Manual," 4 April 2014. [Online]. Available: http://download.lulzbot.com/TAZ/4.0/documentation/2014Q2/manual/TAZ\_4\_Manual.pdf.
- [11] C. Xu, "Multifunctional Ceramic Matrix Nanocomposites Manufacturing," 2014.

- [12] D. o. H. a. H. Services, "Center for Desease Control and Prevention," 2010. [Online]. Available: http://www.cdc.gov/niosh/docs/2013-145/pdfs/2013-145.pdf. [Accessed 8th April 2015].
- [13] D. Company, "DNF Solution," 2 April 2008. [Online]. Available: http://www.dnfsolution.com/MSDS/06\_Gap\_Fill\_Material/01\_DNF-PS%28eng%29%20MSDS.pdf. [Accessed 8 April 2015].
- [14] R. Drew and J. Frangos, "Engineered nanomaterials: a review of the toxicology and health hazards," Toxikos Pty Ltd., Canberra, 2009.
- [15] CSIRO, "Safe Handling and Use of Carbon Nanotubes," Safe Work Australia, Canberra.
- [16] University of California Irvine, "Ultraviolet Lamp Safety Factsheet," [Online]. Available: https://www.ehs.uci.edu/programs/radiation/UV%20Lamp%20Safety%20Factsheet.pdf. [Accessed 29 March 2015].
- [17] P. Byszewski and M. Baran, "Magnetic Susceptibility of Carbon Nanotubes," *Europhysics Letters*, vol. 31, no. 7, 1995.

# Appendix A. TAZ 4 Safety Warnings

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http://download.lulzbot.com/TAZ/4.0/documentation/2014Q2/manual/TAZ\_4\_Manual.pdf

#### WARNING!

#### Read Me First!

# READ THIS MANUAL COMPLETELY BEFORE UNPACKING AND POWERING UP YOUR PRINTER.

#### Hazards and Warnings

The TAZ 3D printer has motorized and heated parts. Always be aware of possible hazards when the printer is operational.

#### Electric Shock Hazard

Never open the electronics case when the printer is powered on. Before removing the electronics case cover always power down the printer and completely turn off and unplug the power supply. Allow the power supply to discharge for at least one minute.

#### Burn Hazard

Never touch the extruder nozzle or heater block without first turning off the hot end and allowing it to completely cool down. The hot end can take up to 20 minutes to completely cool. Never touch recently extruded plastic. The plastic can stick to your skin and cause burns. The heated bed can reach high temperatures that are capable of causing burns.

#### Fire Hazard

Never place flammable materials or liquids on or near the printer when it is powered on or operational. Liquid acetone and vapors are extremely flammable.

#### Pinch Hazard

When the printer is operational take care to never put your fingers in any moving parts including belts, pulleys, or gears. Tie back long hair or clothing that can get caught in the moving parts of the printer.

#### HAZARDS AND WARNINGS

#### Static Charge

Make sure to ground yourself before touching the printer, especially its electronics. Electrostatic discharge can damage electronic components. Ground yourself by touching a grounded source like the metal power supply housing or your computer case.

#### Age Warning

For users under the age of 18, adult supervision is recommended. Beware of choking hazards around small children.

## Appendix B. Syringe.m MATLAB Code

%Syringe Pressure
F\_1 = 15; %linear force of syringe pump at low speeds (lb)
F\_2 = 35; %linear force of syringe pump at top speeds (lb)
F\_all = [15, 17, 19, 21, 23, 25, 27, 30, 35]; %low to high speeds

\*\*\*

% Needle gauges 18, 20, 22, 30
% 18 gauge inside diameter = 0.838mm
% 20 gauge ID = 0.603mm
% 22 gauge ID - 0.400mm
% 30 gauge ID = 0.156mm

#### *୫*୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫

% Pressure of pump for 20mL syringe % 60mL ID is 26.7 mm d = 0.8543; %inside diameter of syringe in in A = (pi/4)\*(d^2); % area in inches squared of ID P\_lowspeed = (F\_1/A)\*6.895 %Pressure of pump at low speeds in kPa P\_topspeed = (F\_2/A)\*6.895 %Pressure of pump at top speeds in kPa P\_all = (F\_all/A) \*6.895 % Pressure of pump at varying speeds in kPa

#### 

%Pressures for only the needle length %assuming barrel ID is the diameter of needle gauge d\_var = [0.006142, 0.01575, 0.02374, 0.03299]; %ID of needles in inches % in order from 30 decreasing down to 18 gauge  $A_var = (pi/4)*(d_var.^2);$  $A_400 = (pi/4)*(0.01575^2);$ l\_total = 5.325; % total length of syringe in inches %assuming using 1/2" needles 1 = 0.5/5.325; %length of needle divided by total length P\_lowspeed = ((F\_1./A\_var)\*1); %pressure of pump at low speeds in psi P\_topspeed = ((F\_2./A\_var)\*1); % pressure of pump at top speeds in psi P\_all=((F\_all/A\_400)\*1);%400micro pressure of pump at varying speeds in psi % 1psi = 6.895kPa P\_lowspeed = P\_lowspeed \* 6.895 % pressure of pump at top speeds kPa P topspeed = P topspeed \* 6.895 % pressure of pump at low speeds kPa P\_all = P\_all \* 6.895 % pressure of 400 micron kPa

#### \*\*\*

```
% Volume fraction of CNT
%Volume = mass/density
m_p = 0.010; % mass of polysilazane in (kilograms)
rho_p = 1018.525; % density of polysilazane (kg/m^3)
m_cnt = 0.0002; % mass of CNT in (kilograms)
rho_cnt = 2100; %denisty of multiwalled CNT (kg/m^3)
V_p = m_p/rho_p; %volume of polysilazane
V_cnt = m_cnt/rho_cnt; %volume of cnt
```

phi\_p = V\_p/(V\_p + V\_cnt) % Volume fraction for polysilazane
phi\_cnt = V\_cnt/(V\_p + V\_cnt) % Volume fraction for CNT

#### *୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫୫*୫୫୫୫

% Viscosity of Slurry %mu\_p = [50, 200]; %viscosity of polysilazane in (cps) or (mPas) %rho\_p = 1018.525; % density of polysilazane (kg/m^3) %rho\_cnt = 2100; %denisty of multiwalled CNT (kg/m^3) %phi\_cnt =0.0096; %mu\_slurry = mu\_p \* (1+2.5\*(phi\_cnt))%%%????????????

#### 

% Flow Rate from programmable syringe pump q\_1 = 1260 % max flow rate for 30mL syringe (mL/hr) q\_2 = 0.01732 % minimum flow rate for 30mL syringe (mL/hr) q\_1/60 %max flow rate in mL/min q\_2/60 %minimum flow rate in mL/min % tests were ran at 0.1 - 3.0 mL/min q\_max = q\_1/60; %max flow rate mL/min q = (q\_max .\* 0.001)/(60\*1000); % (m^3/s) volumetric flowrate q\_act = (0.5 \*0.001)/(60\*1000) % 0.5mL to m^3/s

#### \*\*\*

% Velocity of fluid q\_act = (0.5 \*0.001)/(60\*1000); % 0.5mL to m^3/s %velocity of fluid = volumetric flow rate/ area d = 0.01575; %diameter of 400 micrometer needle in (m) A = (pi/4)\*(d^2); velocity = q\_act / A % velocity of 400 micro meter needle (m/s) q\_var = [0.1, 0.3, 0.5, 0.7, 1.0, 2.0, 3.0, 4.0, 5.0]; %mL/min q\_var1 = (q\_var.\* 0.001)/(60\*1000) %0.1mL/min - 5mL/min

#### 

%Volumetric Flow Rate using Poiseuille Law for barrel ID w/o needles %Q = (pi\*Pressure difference\*r^4)/(8\*viscosity\*length) r = (d\_var/2) .\* 0.0254; %radius of needles r\_1 = (d/2)\*0.0254; % radius of 400 micorn needle delta\_P = P\_lowspeed - 101.325; % pressure loss, atm pressure = 101.325kPa Pressure\_drop = 49850 - 101.325; %pressure of 400 micron - atm L = 0.5 \* 0.0254; vis = (pi .\* delta\_P .\* r.^4)/(8 \* q\_act \* L) %in kPas vis\_var = (pi \* Pressure\_drop \* r\_1.^4)./(8 \* q\_var1 \* L); %in kPas vis\_var = vis\_var .\*1000 %viscoity with changing flow rate 400 micron

#### \*\*\*

% Diameter of droplet/ line relationship with Re number % Re = density \* velocity \* diameter(or length) / absolute viscosity % flow is laminar for pipe % Reynold number for laminar pipe flow < 2300</pre>

```
Re = (rho_cnt * velocity .* d_var)./ vis %Re at 0.5 mL/min
Re_1 = (rho_cnt * velocity * d)./ vis_var
% Re << 1 Stokes flow occurs
%inertial forces are small compared to viscous forces
rho_slurry = (Re .* vis)/(velocity .* d_var);
vis = vis*1000;
Dia = (Re_1 .* vis_var)./(rho_cnt * velocity); %diameter of droplet
%changing flow rate does not change diameter
figure(1);
plot(q_var1,vis_var);
xlabel('volumetric flow rate (m^3/s)');
ylabel('viscosity (Pa*s)');
figure(2);
plot(Re_1,q_var1);
xlabel ('Reynolds #');
ylabel (' Volumetric Flow Rate (m^3/s)');
```

# Appendix C. Subcomponent Specifications Sheets

# C1 Logitech C920 HD Webcam

General Product Information	[Compliance Certification (CE) Link]				
Warranty / Self Help	Please see product support page for warranty duration and frequently asked questions.				
Category	Webcam				
Software Support (at release)	Logitech Webcam Software Version: 2.4				
	NOTE: If software is available, check website for latest software release.				
OS Support (at release)	Windows XP x32 / x64, Windows Vista x32 / x64, Windows 7 x32 / x64				
	Basic Requirement	HD Requirement			
System Requirements	CPU Minimum = 1.0 Ghz CPU Recommended = Core 2 Duo 2.4Ghz or better RAM Minimum = 256 PAM Percommended =	CPU Minimum = Core 2 Duo 2.4Ghz or better CPU Recommended = i7 Quad Core 2.6Ghz or better RAM Minimum = 2GB			
	2GB	RAM Recommended =4GB			
General Product Information	2GB [Compliance Cer	RAM Recommended =4GB			
General Product Information Warranty / Self Help	2GB [Compliance Cen Please see product support frequently a	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions.			
General Product Information Warranty / Self Help Category	2GB [Compliance Cen Please see product support frequently a We	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions. ebcam			
General Product Information Warranty / Self Help Category	Please see product support frequently a United – 2GB	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions. ebcam Software Version: 2.4			
General Product Information Warranty / Self Help Category Software Support (at release)	Compliance Cer Please see product support frequently a Wa Logitech Webcam NOTE: If software is available re	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions. ebcam . Software Version: 2.4 e, check website for latest software elease.			
General Product Information Warranty / Self Help Category Software Support (at release) OS Support (at release)	In the commended – 2GB I Compliance Cer Please see product support frequently a Wo Logitech Webcam NOTE: If software is available re Windows XP x32 / x64, Windows x3	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions. ebcam . Software Version: 2.4 e, check website for latest software elease. dows Vista x32 / x64, Windows 7 2 / x64			
General Product Information Warranty / Self Help Category Software Support (at release) OS Support (at release)	ICompliance Cer Please see product support frequently a Unit Logitech Webcam NOTE: If software is available ref Windows XP x32 / x64, Windows XBasic Requirement	RAM Recommended =4GB rtification (CE) Link] page for warranty duration and asked questions. ebcam Software Version: 2.4 e, check website for latest software elease. dows Vista x32 / x64, Windows 7 2 / x64 HD Requirement			

RAM Minimum = 256 RAM Recommended = 2GB Core 2.6Ghz or better RAM Minimum = 2GB RAM Recommended =4GB

Webcam Specification	ons					
<b>Connection Type</b>		USB				
<b>USB</b> Protocol		USB 2.0				
USB VID_PID		082D				
UVC Support		Yes				
Microphone		Yes				
Microphone Type		Stereo				
Lens and Sensor Ty	ype	Glass				
Focus Type		Auto				
<b>Optical Resolution</b>		True:3MP Software Enhanced:15MP				
Diagonal Field of V (FOV)	ïew	78°				
Focal Length		3.67 mm				
Image Capture (16)	:9 W)	2.0 MP, 3 MP*, 6 MP*, 15 MP*				
Video Capture (16:	9 W)	360p, 480p, 720p, 1080p				
Frame Rate (max)		1080p@30fps				
Right Light		RightLight 2				
Video Effects (VFX	.)	N/A				
Buttons		N/A				
Indicator Lights (L	ED)	Yes				
Privacy Shade		No				
Tripod Mounting (	Option	Yes				
Cable Length		6 feet				
Product Dimensions						
Product component	Width	Depth/Length He	eight	Weight		
Webcam	94 mm	24 mm 29	mm	162g		

# C2 Melody Susie UV Nail Dryer

http://www.melodysusie.com/products/product\_detail/195

Melody Susie - Violeteer UV Nail Dryer -

Product Details: Color: black

- Auto timer control: 120s, 180s, up to infinite

- Material: Molded ABS

- 5\*9W 365nm UV Bulb

Package Includes:

- 1\* Melody Susie UV Nail Dryer (Black, 36W)
- 5\* 9W 365nm UV Bulb
- 1 \* Manual
- **Product Details**

Product Dimensions: 9.4 x 8.3 x 3.9 inches; 2.2 pounds

Shipping Weight: 2.7 pounds (View shipping rates and policies)

# C3 Etekcity 630 Digital Dual Laser Infrared Thermometer



Features:

- Dual laser targeting system
- Narrow distance-spot ratio offers accurate results at greater distances
- Safely measure hazardous or inaccessible objects
- Standard 9V battery offers 14 hours of cumulative use
- Auto-shutoff after 15 seconds to increase battery life
- Instant-read-displays results in less than one second.
Appendix D. Component Drawings

D1 Syringe Pump Exploded View with Integrated Curing System





# D2 Syringe Pump Assembly View

# D3 Curing Fixture



# D4 End Plate



## Appendix E. Reliability Calculations

- $R(t) \rightarrow Reliability at time t$
- $\lambda \rightarrow Failure Rate$

•

- $MTBF \rightarrow Mean Time Before Failure$
- **Data:** R = 76.5%, t = 5 hours/print job

Calculation #1: Failure Rate

$$R(t) = e^{-\lambda t}$$
$$\ln(0.765) = -5\lambda$$

 $\lambda \Rightarrow 0.0536 \ failures/hour$ 

 $\lambda \approx 5.36$  failures every 100 hours

Calculation #2: Mean Time Before Failure

$$MTBF = \frac{1}{\lambda}$$
$$MTBF \approx \frac{1}{0.0536}$$

 $MTBF \approx 18.66 hrs$ 

 $18.66 hrs \cong 4 print jobs$ 

# Appendix F. Total Allocated Project Costs

ITEM	TOTAL COST
ARDUINO MEGA	\$45.95
INKSHIELD	\$66.00
UV LIGHTS	\$58.06
<b>3D PRINTER</b>	\$1,995.00
NEEDLES	\$83.30
THERMOMETER	\$29.99
WEBCAM	\$69.65
TRIPOD	\$22.75
ABS FILAMENT	\$171.80
UV LAMP	\$89.00
UV SAFETY GLASSES	\$74.24
SYRINGES	\$9.50
<b>BLUNT TIPS</b>	\$9.90
CARTRIDGES	\$19.98
NEEDLE KIT	\$19.93
NOZZLE	\$19.00
<b>BLUNT TIPS 22</b>	\$12.99
CRIMP-ON	\$5.35
INSULATED DISCONNECTS	\$2.99
HOOK UP WIRE	\$8.56
STEEL SHEET	\$12.63
<b>USB ADAPTER</b>	\$37.60
DC POWER ADAPTER	\$5.35
AC ADAPTER	\$13.80
FEMALE ADAPTER	\$4.99
CONN PLUG	\$4.48
THREADED EXTENSION	\$11.00
HEATER BLOCK	\$15.00
TAXES/SHIPPING	\$19.46
TOTAL	2,891.60

# Appendix G. Material Safety Data Sheets

# G1 Polymer Precursor

The data sheet is reprinted on the following page.



Revision Date: February 2, 2005

### **KiON<sup>®</sup> Corporation**

1957-A Pioneer Road, Huntingdon Valley, Pennsylvania 19006 Phone: 1-215-957-6100 Fax: 1-215-957-6324

24-Hour Emergency Phone: CHEMTREC (USA) 1-800-424-9300, (International) 1-703-527-3887

### MATERIAL SAFETY DATA SHEET

SECTION 1 Chemical Product Identification

NAME:

KiON<sup>®</sup> Ceraset<sup>®</sup> Polysilazane 20

PRODUCT CODE: C-2001

### SECTION 2 Composition/Information on Ingredients

COMPOSITION:

CAS No:503590-70-3Chemical Name:"Cyclosilazanes, methyl hydrogen, methyl vinyl"Chemical Identity:PolysilazanePercent:>99 %

### SECTION 3 Hazards Identification

HMIS RATINGS

Health hazard	3	Serious
Flammability hazard	3	Serious
Reactivity hazard	1	Slight

### POTENTIAL HEALTH EFFECTS:

Signs and Symptoms of Overexposure in the Workplace:

WARNING: Corrosive, Flammable liquid May cause severe eye irritation or eye burns. Causes skin burns with redness, pain and swelling. May cause irritation of mucous membranes if vapor or mist is inhaled.

Medical Conditions Aggravated by Exposure: None known

Primary Routes of Exposure/Entry:

Page 1 of 5

Product Code: C-2001

Eye or skin contact; inhalation of dust. Ingestion unlikely. Note: Contact of this product with moisture can produce ammonia, which can cause irritation of the eyes, skin, nose, throat, lungs, etc.

### SECTION 4 First-Aid Measures

### EYE EXPOSURE:

In case of contact with eyes immediately flush with copious amounts of water for at least 15 minutes. If irritation or redness persists seek medical treatment.

### DERMAL EXPOSURE:

In case of contact with skin or clothing; remove contaminated clothing and wash skin thoroughly with soap and water. If irritation or redness persists seek medical treatment.

### INHALATION EXPOSURE:

If inhaled, remove to fresh air. If breathing is difficult, give oxygen and seek immediate medical assistance. If not breathing give artificial respiration and seek immediate medical assistance.

### ORAL EXPOSURE:

If swallowed do not induce vomiting. Seek immediate medical treatment.

### SECTION 5 Fire Fighting Measures

EXTINGUISHING MEDIA: Carbon dioxide, dry chemical powder or appropriate foam.

### SPECIAL FIREFIGHTING PROCEDURES:

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.

Use water spray to cool fire-exposed containers.

UNUSUAL FIRE AND EXPLOSIONS HAZARDS: Container explosion may occur under fire conditions. May emit toxic fumes under fire conditions.

### SECTION 6 Accidental Release Measures

For small spills: Cover with an inorganic absorbent, like vermiculite, perlite, ground clay, or sand, sweep up, and dispose appropriately.

Page 2 of 5

Product Code: C-2001

For large spills: Dike to contain and pump into drums for use or disposal. If any material remains add inorganic absorbent (as above), sweep up, and dispose appropriately. Clean contaminated area with soap and water. In case of accidental spill or release, refer to Section 8, Personal Protective Equipment and General Hygiene Practices.

### SECTION 7 Handling and Storage

Flammable. Store in areas designated for flammable liquid storage (see NFPA requirements). Keep away from heat, sparks, and open flame. Keep tightly closed. Vent periodically to release head pressure.

### SECTION 8 Exposure Controls/Personal Protection

### APPROPRIATE HYGIENIC PRACTICES:

Avoid contact with eyes, skin, and clothing. Avoid breathing vapors, fumes and mists. Avoid prolonged or repeated exposure. Wash thoroughly after handling, and before eating, drinking, or smoking.

### ENGINEERING CONTROLS:

Engineering controls should always be used when available as a first choice over personal protective equipment. Provide adequate ventilation. Use of fume hoods or closed booths recommended when product is used in a manner that may generate mist or aerosol.

### PERSONAL PROTECTIVE EQUIPMENT:

Normal laboratory protective clothing recommended, i.e. lab coat and/or apron, impervious gloves and safety glasses. If mists or aerosols are generated during handling, and engineering controls are not present to prevent exposure, wear chemical safety goggles and a respirator equipped with and organic vapor cartridge.

### WORK PRACTICES:

Easily accessible eyewash fountains and safety showers recommended.

### PROTECTIVE MEASURES DURING REPAIR AND MAINTENANCE:

Completely isolate and thoroughly clean all equipment, piping or vessels with high flash non-polar solvents before beginning maintenance or repairs.

### SECTION 9 Physical and Chemical Properties

APPEARANCE AND ODOR: Liquid, clear to pale yellow. May detect slight odor of ammonia immediately after opening container.

Page 3 of 5

Product Code: C-2001

PHYSICAL PROPERTIES:

Not applicable, material slowly cross-links to a solid upon heating.
Not Determined (note: a similar material had a flash point of 29°C
(84°F) closed cup)
1.0
184 cps at 19°C (67°F) nominal
Not Determined

### SECTION 10 Stability and Reactivity

STABILITY: Stable

INCOMPATIBILITIES:

Reacts vigorously and exothermically with isocyanates. Material will react slowly with water and other protic solvents. This product may react with mineral acids, alkalies, and oxidizing agents. Caution should be taken when mixing this product with any of these materials.

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Carbon monoxide, Carbon dioxide, Silicon dioxide

HAZARDOUS POLYMERIZATION: Will not occur.

### SECTION 11 Toxicological Information

CARCINOGENICITY INFORMATION:

Not listed as a carcinogen by NTP (National Toxicology Program]; not regulated as a carcinogen by OSHA (Occupational Safety and Health Administration); not evaluated by IARC (International Agency for Research on Cancer).

**REPORTED HUMAN EFFECTS:** 

No human studies have been conducted with this material. The use of recommended protective equipment should prevent any adverse effects. The KiON Corporation has not received any reports of adverse effects from workers handling this material.

**REPORTED ANIMAL EFFECTS:** 

Oral LD50, rat: > 2,000 mg/kg. Skin irritation, rabbit: severe erythema with signs of necrosis after 1-hour exposure.

### SECTION 12 Ecological Information

This material may be hazardous to aquatic organisms. Avoid release to surface waters and waste treatment systems.

Page 4 of 5

MATERIAL SAFETY DATA SHEET - KION<sup>®</sup> Ceraset<sup>®</sup> Polysilazane 20

Product Code: C-2001

### SECTION 13 Disposal Considerations

Do not mix this product with aqueous or other protic wastes streams. Incineration of combustible waste material in a permitted facility in accordance with the local, state, and federal regulations is the recommended disposal method.

### SECTION 14 Transportation Information

US DOT:	Corrosive liquid, flammable
IATA:	Corrosive liquid, flammable, n.o.s., (polysilazane), 8.3, UN2920, II

### SECTION 15 Regulatory Information

SARA 313: This product does not contain any chemicals subject to reporting under Section 313 of Title III of the Superfund Amendments and Reauthorization Act and 40CFR372.

CERCLA: This product does not contain any chemicals subject to reporting as a CERCLA Hazardous Substance under 40CFR302.4.

TSCA: This product is TSCA listed and Compliant under EPA Accession# P88-1778.

- EU Labeling
  - C: Corrosive
  - F: Flammable
- EU Risk and Safety Phrases:
  - R10: Flammable
  - R35: Causes severe skin burns.
  - S16: Keep away from sources of ignition. No smoking.
  - S23: Do not breathe gas/fumes/vapor/spray.
  - S24/25/26: Avoid contact with skin and eyes. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
  - S28: After contact with skin, wash immediately with plenty of water.
  - S37/39: Wear suitable gloves and eye/face protection.

### SECTION 16 Other Information

The above information is believed to be correct but does not purport to be all-inclusive and shall be used only as a guide. KiON Corporation shall not be held liable for any damage resulting from handling or from contact with the above product.

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# G2 Acetone





### Material Safety Data Sheet Acetone MSDS

Product Name: Acetone

Catalog Codes: SLA3502, SLA1645, SLA3151, SLA3808

CAS#: 67-64-1

RTECS: AL3150000

TSCA: TSCA 8(b) inventory: Acetone

CI#: Not applicable.

**Synonym:** 2-propanone; Dimethyl Ketone; Dimethylformaldehyde; Pyroacetic Acid

### Chemical Name: Acetone

Chemical Formula: C3-H6-O

Contact Information:

Sciencelab.com, Inc. 14025 Smith Rd. Houston, Texas 77396

US Sales: 1-800-901-7247 International Sales: 1-281-441-4400 Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

### Section 2: Composition and Information on Ingredients

### Composition:

Name	CAS #	% by Weight
Acetone	67-64-1	100

Toxicological Data on Ingredients: Acetone: ORAL (LD50): Acute: 5800 mg/kg [Rat]. 3000 mg/kg [Mouse]. 5340 mg/kg [Rabbit]. VAPOR (LC50): Acute: 50100 mg/m 8 hours [Rat]. 44000 mg/m 4 hours [Mouse].

### Section 3: Hazards Identification

### Potential Acute Health Effects:

Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

### Potential Chronic Health Effects:

CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female, Reproductive system/toxin/male [SUSPECTED]. The substance is toxic to central nervous system (CNS). The substance may be toxic to kidneys, the reproductive system, liver, skin. Repeated or prolonged exposure to the substance can produce target organs damage.

### Section 4: First Aid Measures

p. 1

### Eye Contact:

Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Get medical attention.

### Skin Contact:

In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

### Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek medical attention.

### Inhalation:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if symptoms appear.

### Serious Inhalation:

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

### Ingestion:

Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention if symptoms appear.

Serious Ingestion: Not available.

### Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 465°C (869°F)

Flash Points: CLOSED CUP: -20°C (-4°F). OPEN CUP: -9°C (15.8°F) (Cleveland).

Flammable Limits: LOWER: 2.6% UPPER: 12.8%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances: Highly flammable in presence of open flames and sparks, of heat.

### Explosion Hazards in Presence of Various Substances:

Risks of explosion of the product in presence of mechanical impact: Not available. Slightly explosive in presence of open flames and sparks, of oxidizing materials, of acids.

### Fire Fighting Media and Instructions:

Flammable liquid, soluble or dispersed in water. SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use alcohol foam, water spray or fog.

Special Remarks on Fire Hazards: Vapor may travel considerable distance to source of ignition and flash back.

### Special Remarks on Explosion Hazards:

Forms explosive mixtures with hydrogen peroxide, acetic acid, nitric acid, nitric acid + sulfuric acid, chromic anydride, chromyl chloride, nitrosyl chloride, nitrosyl perchlorate, nitryl perchlorate, permonosulfuric acid, thiodiglycol + hydrogen peroxide, potassium ter-butoxide, sulfur dichloride, 1-methyl-1,3-butadiene, bromoform, carbon, air, chloroform, thitriazylperchlorate.

### Section 6: Accidental Release Measures

### Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container.

### Large Spill:

Flammable liquid. Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

### Section 7: Handling and Storage

### Precautions:

Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, reducing agents, acids, alkalis.

### Storage:

Store in a segregated and approved area (flammables area). Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Keep away from direct sunlight and heat and avoid all possible sources of ignition (spark or flame).

### Section 8: Exposure Controls/Personal Protection

### **Engineering Controls:**

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

### Personal Protection:

Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

### Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

### Exposure Limits:

TWA: 500 STEL: 750 (ppm) from ACGIH (TLV) [United States] TWA: 750 STEL: 1000 (ppm) from OSHA (PEL) [United States] TWA: 500 STEL: 1000 [Austalia] TWA: 1185 STEL: 2375 (mg/m3) [Australia] TWA: 750 STEL: 1500 (ppm) [United Kingdom (UK)] TWA: 1810 STEL: 3620 (mg/m3) [United Kingdom (UK)] TWA: 1800 STEL: 2400 from OSHA (PEL) [United States]Consult local authorities for acceptable exposure limits.

### **Section 9: Physical and Chemical Properties**

Physical state and appearance: Liquid.

Odor: Fruity. Mint-like. Fragrant. Ethereal

Taste: Pungent, Sweetish

Molecular Weight: 58.08 g/mole

Color: Colorless. Clear

pH (1% soln/water): Not available.

**Boiling Point:** 56.2°C (133.2°F)

Melting Point: -95.35 (-139.6°F)

Critical Temperature: 235°C (455°F)

Specific Gravity: 0.79 (Water = 1)

Vapor Pressure: 24 kPa (@ 20°C) Vapor Density: 2 (Air = 1) Volatility: Not available. Odor Threshold: 62 ppm Water/Oil Dist. Coeff.: The product is more soluble in water; log(oil/water) = -0.2 Ionicity (in Water): Not available. Dispersion Properties: See solubility in water.

Solubility: Easily soluble in cold water, hot water.

### Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Excess heat, ignition sources, exposure to moisture, air, or water, incompatible materials.

Incompatibility with various substances: Reactive with oxidizing agents, reducing agents, acids, alkalis.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

### Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.

#### Toxicity to Animals:

WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE. Acute oral toxicity (LD50): 3000 mg/kg [Mouse]. Acute toxicity of the vapor (LC50): 44000 mg/m3 4 hours [Mouse].

### **Chronic Effects on Humans:**

CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH. DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female, Reproductive system/toxin/male [SUSPECTED]. Causes damage to the following organs: central nervous system (CNS). May cause damage to the following organs: kidneys, the reproductive system, liver, skin.

### Other Toxic Effects on Humans:

Hazardous in case of skin contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

### Special Remarks on Toxicity to Animals: Not available.

### Special Remarks on Chronic Effects on Humans:

May affect genetic material (mutagenicity) based on studies with yeast (S. cerevisiae), bacteria, and hamster fibroblast cells. May cause reproductive effects (fertility) based upon animal studies. May contain trace amounts of benzene and formaldehyde which may cancer and birth defects. Human: passes the placental barrier.

### Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects: Skin: May cause skin irritation. May be harmful if absorbed through the skin. Eyes: Causes eye irritation, characterized by a burning sensation, redness, tearing, inflammation, and possible corneal injury. Inhalation: Inhalation at high concentrations affects the sense organs, brain and causes respiratory tract irritation. It also may affect the Central Nervous System (behavior) characterized by dizzness, drowsiness, confusion, headache, muscle weakeness, and possibly motor incoordination, speech abnormalities, narcotic effects and coma. Inhalation may also affect the gastrointestinal tract (nausea, vomiting). Ingestion: May cause irritation of the digestive (gastrointestinal) tract (nausea, vomiting). It may also

affect the Central Nevous System (behavior), characterized by depression, fatigue, excitement, stupor, coma, headache, altered sleep time, ataxia, tremors as well at the blood, liver, and urinary system (kidney, bladder, ureter) and endocrine system. May also have musculoskeletal effects. Chronic Potential Health Effects: Skin: May cause dermatitis. Eyes: Eye irritation.

### Section 12: Ecological Information

### Ecotoxicity:

Ecotoxicity in water (LC50): 5540 mg/l 96 hours [Trout]. 8300 mg/l 96 hours [Bluegill]. 7500 mg/l 96 hours [Fatthead Minnow]. 0.1 ppm any hours [Water flea].

BOD5 and COD: Not available.

### Products of Biodegradation:

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The product itself and its products of degradation are not toxic.

Special Remarks on the Products of Biodegradation: Not available.

### Section 13: Disposal Considerations

#### Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

### Section 14: Transport Information

DOT Classification: CLASS 3: Flammable liquid.

Identification: : Acetone UNNA: 1090 PG: II

### Special Provisions for Transport: Not available.

### Section 15: Other Regulatory Information

### Federal and State Regulations:

California prop. 65: This product contains the following ingredients for which the State of California has found to cause reproductive harm (male) which would require a warning under the statute: Benzene California prop. 65: This product contains the following ingredients for which the State of California has found to cause birth defects which would require a warning under the statute: Benzene California prop. 65: This product contains the following ingredients for which the State of California has found to cause birth defects which would require a warning under the statute: Benzene California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Benzene, Formaldehyde Connecticut hazardous material survey.: Acetone Illinois toxic substances disclosure to employee act: Acetone Illinois chemical safety act: Acetone New York release reporting list: Acetone Rhode Island RTK hazardous substances: Acetone Pennsylvania RTK: Acetone Florida: Acetone Minnesota: Acetone Massachusetts RTK: Acetone Massachusetts spill list: Acetone New Jersey: Acetone Louisiana spill reporting: Acetone California List of Hazardous Substances (8 CCR 339): Acetone TSCA 8(b) inventory: Acetone TSCA 4(a) final test rules: Acetone TSCA 8(a) IUR: Acetone

### **Other Regulations:**

OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

### Other Classifications:

### WHMIS (Canada):

CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F). CLASS D-2B: Material causing other toxic effects (TOXIC).

р. 5

### DSCL (EEC):

R11- Highly flammable. R36- Irritating to eyes. S9- Keep container in a well-ventilated place. S16- Keep away from sources of ignition - No smoking. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 1

Flammability: 3

Reactivity: 0

Specific hazard:

### Protective Equipment:

Gloves. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Splash goggles.

### Section 16: Other Information

### References:

-Material safety data sheet issued by: la Commission de la Santé et de la Sécurité du Travail du Québec. -The Sigma-Aldrich Library of Chemical Safety Data, Edition II. -Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987. LOLI, RTECS, HSDB databases. Other MSDSs

Other Special Considerations: Not available.

Created: 10/10/2005 08:13 PM

Last Updated: 05/21/2013 12:00 PM

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# G3 Single Walled Carbon Nanotubes

## SIGMA-ALDRICH

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### SAFETY DATA SHEET

Version 5.4 Revision Date 07/01/2014 Print Date 03/29/2015

1. PF	RODUCT AND COMPANY IDEN	ITIFICATION	
1.1	<b>Product identifiers</b> Product name :	Carbon nanotube, single-walled	
	Product Number : Brand :	704113 Aldrich	
	CAS-No. :	308068-56-6	
1.2	.2 Relevant identified uses of the substance or mixture and uses advised against		
	Identified uses	Laboratory chemicals, Manufacture of substances	
1.3	1.3 Details of the supplier of the safety data sheet		
	Company :	Sigma-Aldrich 3050 Spruce Street SAINT LOUIS MO 63103 USA	
	Telephone : Fax :	+1 800-325-5832 +1 800-325-5052	
1.4	Emergency telephone numb	er	
	Emergency Phone # :	(314) 776-6555	
2. HA	ZARDS IDENTIFICATION		
2.1	.1 Classification of the substance or mixture		
	<b>GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)</b> Eye irritation (Category 2A), H319 Specific target organ toxicity - single exposure (Category 3), Respiratory system, H335		
	For the full text of the H-Statements mentioned in this Section, see Section 16.		
2.2	GHS Label elements, includ	ing precautionary statements	
	Pictogram		
	Signal word	Warning	
	Hazard statement(s) H319 H335	Causes serious eye irritation. May cause respiratory irritation.	
	Precautionary statement(s) P261 P264 P271 P280 P304 + P340 P305 + P351 + P338 P312 P337 + P313	Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray. Wash skin thoroughly after handling. Use only outdoors or in a well-ventilated area. Wear protective gloves/ eye protection/ face protection. IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Call a POISON CENTER or doctor/ physician if you feel unwell. If eye irritation persists: Get medical advice/ attention.	

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P403 + P233	Store in a well-ventilated place. Keep container tightly closed.
P405	Store locked up.
P501	Dispose of contents/ container to an approved waste disposal plant

### 2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

### **3. COMPOSITION/INFORMATION ON INGREDIENTS**

### 3.1 Substances

Synonyms	:	Carbon Nanotubes		
CAS-No.	:	308068-56-6		
Hazardous components				
Component			Classification	Concentration
Carbon Nanotubes				
			Eye Irrit. 2A; STOT SE 3; H319, H335	-

For the full text of the H-Statements mentioned in this Section, see Section 16.

### 4. FIRST AID MEASURES

### 4.1 Description of first aid measures

### General advice

Consult a physician. Show this safety data sheet to the doctor in attendance. Move out of dangerous area.

### lf inhaled

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

### In case of skin contact

Wash off with soap and plenty of water. Consult a physician.

### In case of eye contact

Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

#### If swallowed

Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

### 4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

## 4.3 Indication of any immediate medical attention and special treatment needed no data available

### **5. FIREFIGHTING MEASURES**

### 5.1 Extinguishing media

#### Suitable extinguishing media

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

## 5.2 Special hazards arising from the substance or mixture Carbon oxides

#### 5.3 Advice for firefighters Wear self contained breathing apparatus for fire fighting if necessary.

- 5.4 Further information
  - no data available

### 6. ACCIDENTAL RELEASE MEASURES

### 6.1 Personal precautions, protective equipment and emergency procedures

Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust. For personal protection see section 8.

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### 6.2 Environmental precautions

Do not let product enter drains.

### 6.3 Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

### 6.4 Reference to other sections

For disposal see section 13.

### 7. HANDLING AND STORAGE

### 7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid formation of dust and aerosols. Provide appropriate exhaust ventilation at places where dust is formed.Normal measures for preventive fire protection. For precautions see section 2.2.

## 7.2 Conditions for safe storage, including any incompatibilities Keep container tightly closed in a dry and well-ventilated place.

#### 7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

### 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

### 8.1 Control parameters

### Components with workplace control parameters

Contains no substances with occupational exposure limit values.

### 8.2 Exposure controls

### Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

### Personal protective equipment

### Eye/face protection

Safety glasses with side-shields conforming to EN166 Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

### Skin protection

For any handling steps where the substance is in particulate form or in a suspension with pure water where the substance is not solubilized, the gloves must be comprised of material that successfully passes ASTM F-1671. For any handling steps where the substance is part of a carrier liquid, other than the aqueous suspension noted in the previous paragraph, gloves must be comprised of material that successfully passes ASTM F-739 (continuous liquid contact method). Gloves must be changed before they show degradation and before the designated breakthrough time for the carrier liquid (as determined by the ASTM F-739 testing or by the manufacturer).

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

### **Body Protection**

impervious clothing, The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

### **Respiratory protection**

The EPA mandates the use of full face respirators with minimum N100 grade cartridges if there is any risk of exposure to carbon nanotube dust.

For nuisance exposures use type P95 (US) or type P1 (EU EN 143) particle respirator. For higher level protection use type OV/AG/P99 (US) or type ABEK-P2 (EU EN 143) respirator cartridges. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

### Control of environmental exposure

Do not let product enter drains.

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### 9. PHYSICAL AND CHEMICAL PROPERTIES

### 9.1 Information on basic physical and chemical properties

a)	Appearance	Form: solid
b)	Odour	no data available
c)	Odour Threshold	no data available
d)	рН	no data available
e)	Melting point/freezing point	3,652 - 3,697 °C (6,606 - 6,687 °F)
f)	Initial boiling point and boiling range	no data available
g)	Flash point	no data available
h)	Evapouration rate	no data available
i)	Flammability (solid, gas)	no data available
j)	Upper/lower flammability or explosive limits	no data available
k)	Vapour pressure	no data available
I)	Vapour density	no data available
m)	Relative density	no data available
n)	Water solubility	insoluble
o)	Partition coefficient: n- octanol/water	no data available
p)	Auto-ignition temperature	no data available
q)	Decomposition temperature	no data available
r)	Viscosity	no data available
s)	Explosive properties	no data available
t)	Oxidizing properties	no data available
Oth	er safety information	

9.2 Other safety inform no data available

### **10. STABILITY AND REACTIVITY**

- 10.1 Reactivity no data available
  10.2 Chemical stability Stable under recommended storage conditions.
- **10.3** Possibility of hazardous reactions no data available
- **10.4 Conditions to avoid** no data available
- **10.5** Incompatible materials Strong oxidizing agents
- **10.6 Hazardous decomposition products** Other decomposition products - no data available In the event of fire: see section 5

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### **11. TOXICOLOGICAL INFORMATION**

### 11.1 Information on toxicological effects

### Acute toxicity

no data available

Inhalation: no data available

Dermal: no data available

no data available

### Skin corrosion/irritation

no data available

### Serious eye damage/eye irritation no data available

### Respiratory or skin sensitisation no data available

Germ cell mutagenicity no data available

### Carcinogenicity

- IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
- ACGIH: No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by ACGIH.
- No component of this product present at levels greater than or equal to 0.1% is identified as a NTP: known or anticipated carcinogen by NTP.
- OSHA: No component of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

#### **Reproductive toxicity** no data available

no data available

Specific target organ toxicity - single exposure Inhalation - May cause respiratory irritation.

### Specific target organ toxicity - repeated exposure no data available

### Aspiration hazard

no data available

### Additional Information

### RTECS: Not available

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

### **12. ECOLOGICAL INFORMATION**

### 12.1 Toxicity

no data available

- 12.2 Persistence and degradability no data available
- 12.3 Bioaccumulative potential no data available

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### **12.4** Mobility in soil no data available

### 12.5 Results of PBT and vPvB assessment

PBT/vPvB assessment not available as chemical safety assessment not required/not conducted

### 12.6 Other adverse effects

no data available

### 13. DISPOSAL CONSIDERATIONS

### 13.1 Waste treatment methods

### Product

Offer surplus and non-recyclable solutions to a licensed disposal company. Contact a licensed professional waste disposal service to dispose of this material.

### Contaminated packaging

Dispose of as unused product.

### 14. TRANSPORT INFORMATION

### DOT (US)

Not dangerous goods

### IMDG

Not dangerous goods

ΙΑΤΑ

Not dangerous goods

### **15. REGULATORY INFORMATION**

### SARA 302 Components

SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

### SARA 313 Components

SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

### SARA 311/312 Hazards

Acute Health Hazard

### Massachusetts Right To Know Components

No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right To Know Components			
Carbon Nanotubes	CAS-No. 308068-56-6	Revision Date	
New Jersey Right To Know Components			
Carbon Nanotubes	CAS-No. 308068-56-6	Revision Date	

### California Prop. 65 Components

This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

### **16. OTHER INFORMATION**

### Full text of H-Statements referred to under sections 2 and 3.

Eye Irrit.	Eye irritation
H319	Causes serious eye irritation.
H335	May cause respiratory irritation
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STOT SE

Specific target organ toxicity - single exposure

### HMIS Rating

Health hazard: 2 Chronic Health Hazard: Flammability: 0 Physical Hazard 0 **NFPA Rating** Health hazard: 2

Health hazard: Fire Hazard: Reactivity Hazard:

0

0

### Further information

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### **Preparation Information**

Sigma-Aldrich Corporation Product Safety – Americas Region 1-800-521-8956

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Revision Date: 07/01/2014

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## Appendix H. Team Biographies

Ernest Etienne - ME

Ernest is a senior at the FAMU-FSU College of Engineering, and is expected to graduate with his B.S. in Mechanical Engineering in May 2015. He is interested in material processing and is working on obtaining his certificate of specialization in Mechanics and Materials.

Cody Evans - IE

Cody is a senior at the FAMU-FSU College of Engineering, and is expected to graduate with his B.S. in Industrial Engineering in May 2015. After graduation he plans on working in industry, and is interested in statistical quality control, project management, and business simulation.

Sonya Peterson - ME

Sonya is an FSU senior mechanical engineering student expecting to graduate in 2015. It is still uncertain where she would like to see herself following graduation, she hopes to progress the movement for a greener future and construct better living standards for every person on the planet to benefit from.

Basak Simal - ME

Basak is an FSU student studying Mechanical Engineering and she will be graduating in Spring 2015. She has not claimed a specialty but has studied thermo-fluids and manufacturing.

Daphne Solis - IE

Daphne is an FSU senior student with a dual major in Industrial Engineering and Actuarial Sciences, and she will be graduating in 2015. She is interested on the fields of quality engineering, industrial systems, and engineering management.

Sam Yang - ME

Samuel is an undergraduate student enrolled at Florida State University, and he will be graduated with a Bachelor of Science in Mechanical Engineering in Spring 2015. He is specializing in the aeronautics field and is a member of the AIAA.