3D PRINT QUALITY IN THE CONTEXT OF PLA COLOR

by
Forest Sears
Candidate BS Materials Science and Engineering,
MIT 2016

Submitted to the Department of Materials Science and Engineering
in partial fulfillment of the requirements for the degree of
Bachelors of Science in Materials Science and Engineering
at the

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Abstract:

3D printing is a hot topic in manufacturing and a truly useful tool, but it has limitations. Print quality properties – like raft peelability, dimensional tolerance and surface roughness – are hard to calibrate perfectly. A common material used in fused deposition modeling (FDM) printers is polylactic acid (PLA). One print quality concern is how different colors of PLA print differently under the exact same settings. The inconsistency in print quality by color is bad for designers, students, and engineers who want to rapidly prototype effectively. Analyzing the thermal, chemical and mechanical properties of the different colors of PLA and relating it to the quality of the prints gives the user a chance to calibrate their machine effectively for higher quality prints. The quality of prints are quantified by scoring systems that measure three properties of a print: dimensional tolerance, how easily the raft peels from the print, and the surface roughness. The thermal properties of the different colors of PLA were analyzed using differential scanning calorimetry (DSC) up to 230°C. The integrals of peaks and troughs from the DSC – representing heat absorbed and released by the different colors of PLA – show that each color responds differently to thermal treatment. The mechanical strength of each color was found to be different through uniaxial tensile testing. Yellow and orange filament had high percent crystallinity at ~12.1%, while having a high yield stress at 41-45 MPa, and a low yield strain at 6.6%-11% extension. Red and blue filament had low percent crystallinity at ~8.8-10.2%, while having a low yield stress at 33-36 MPa, and a high yield strain at 18%-23% extension. Additionally, Fourier transform infrared spectroscopy (FTIR) analysis determined each PLA color had unique additives. For calibrating printers for reliably high quality prints, crystallinity has a relationship with the amount of material extruded which could factor into qualities like dimensional tolerance and surface finish.

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1. Introduction/Background

1.1 Problem Statement

One problem with modern day fused deposition modeling (FDM) 3D printing is that a sample is printed with the same settings regardless if the polylactic acid (PLA) filament is red, white or blue. Currently, all 3D printers don't account for differences in PLA — even though each color of PLA has unique mechanical, chemical, and thermal properties. And unfortunately filament manufacturers want to keep all of their manufacturing techniques and methods quiet and proprietary. Additionally, these manufacturing companies keep the additives for each color of PLA private which would have their own set of unique thermal, chemical and mechanical properties. If the properties of each PLA filament are understood on a deep level this would intelligently motivate the calibration of 3D printers which would mean higher quality prints. Higher quality prints are extremely valuable in the 3D printing space because quality is one of the biggest issues in modern 3D printing. The surface quality of 3D prints are notoriously bad — particularly famous among metal 3D printers — and the resolution isn't always as good as the advertised micron precision.

1.2 Overview of Additive Manufacturing

3D printing is a buzzword that is thrown around a lot in modern technology discussions, specifically in the manufacturing realm. Perhaps a more appropriate word for the concept of 3D printing is the less trendy term, additive manufacturing (AM), but others exist like rapid prototyping, stereolithography or freeform fabrication. AM describes a process where a 3D object is synthesized by forming successive layers of material. AM has been in the market since the 1980s but the AM industry has been making the most noise in the past five years. In 1984, Chuck Hull of 3D systems developed a 3D printer prototype that exposed photopolymers to light causing them to cure — a process that was developed three years before in Japan by Hideo Kodama [1]. So AM isn't a new concept, but it is definitely growing. Today, 3D printing is around a $4.1 billion industry. Compare the current AM industry to the AM market size in 1995, which was only worth around $300 million — or better yet — compare the AM industry to its predicted market size: the projected size of the AM industry is as high as $21.1 billion in 2020 (older reports predict $8.6 billion — which is still a massive industry) [2][3].

Prices of 3D printers vary wildly — a desktop version can be as low as $100 and an industrial version can be as high as $2 million. The higher price of industrial grade printers comes with a lot of capabilities. Industrial printers can print faster at a higher resolution, are more reliable, and are capable of printing in parallel. On the most basic level, typically industrial printers can work with more materials — from polymers to metals to ceramics to composites, whereas most desktop 3D printers work with ABS (acrylonitrile butadiene styrene) and PLA (polylactic acid). The NVPro works mostly with PLA. PLA may be the polymer with the broadest range of applications because of its ability to be stress crystallized, thermally crystallized, impact modified, filled, copolymerized, and processed in most polymer processing equipment [16]. Here's the chemical structure of PLA:

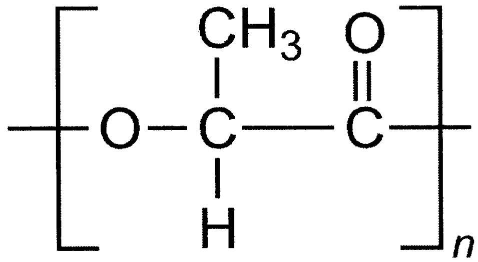


Figure 1. Chemical Structure of PLA. Polylactic acid (PLA) is a biodegradable thermoplastic aliphatic polyester derived from renewable resources, such as corn starch, tapioca roots, chips or starch, or sugarcane.

PLA is perfect for 3D printing because it's a thermoplastic which is strong, lightweight, and transparent (making it easy to dye). PLA is harder than ABS, has a lower glass transition than ABS (~60C vs ~105C), but is more prone to jams because of friction. PLA is also very sustainable relative to other pervasive plastics — PLA's manufacturing process is petroleum free, can be made from corn, potatoes or grains, is recyclable, and is biodegradable. Besides 3D printing, PLA is used for injection molding, investment casting and used in diapers, packaging, and upholstery. PLA is commonly used in fused deposition (FDM) modeling printing which is one of seven types of 3D printing described by the ASTM below [4]:

1. **Binder Jetting** is an additive manufacturing process in which a liquid bonding agent is selectively deposited to join powder materials.
2. **Directed Energy Deposition** is an additive manufacturing process in which focused thermal energy (e.g. laser, electron beam, or plasma arc) is used to fuse materials by melting as they are deposited.
3. **Material Extrusion** is an additive manufacturing process in which material is selectively dispensing through a nozzle or orifice.
4. **Material Jetting** is an additive manufacturing process in which droplets of building materials are selectively deposited.
5. **Powder Bed Fusion** is an additive manufacturing process in which thermal energy selectively fuses regions of a powder bed.
6. **Sheet Lamination** is an additive manufacturing process in which sheets of material are bonded to form an object.
7. **Vat Photopolymerization** is an additive manufacturing process in which liquid photopolymer in a vat is selectively cured by light-activated polymerization.

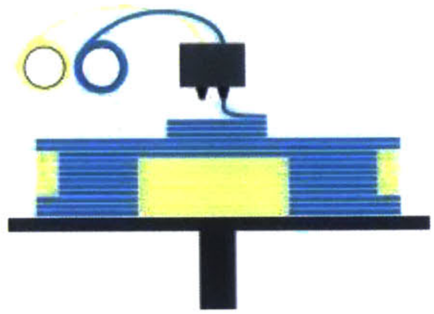


Figure 2. Representation of material extrusion or fused deposition modelling (FDM). The NVBOTS printer is an example of an FDM printer – it extrudes PLA, ABS, or various metals and creates objects layer by layer [12][14].

For desktop and industrial users of any of the seven types, why use 3D printing over traditional manufacturing methods? What makes AM so valuable is how quickly it can speed up reiterating the design, prototyping, and testing process. For industry, this is incredibly valuable in product development. Currently after a product is developed, it needs to be redesigned to make manufacturing as easy, as quick, and as cheap as possible. This is a drawback – a designer should be tailoring a product to a company's users not the company's manufacturing machines and a designer should iterate the design process as many times as possible. At home or at small scale businesses, users don't have to deal with incredibly high upfront costs. AM allows all users to customize a part to specific needs and requirements, mitigate the issue of making complex geometries, be independent of expensive tools or molds, make parts that are sustainable because of material and energy efficiency, and skip tedious steps of the design cycle. And all of this at a price that is dropping rapidly. But AM isn't perfect. There are still issues with reliability, quality, and resolution. 3D printing is lacking the high quality surface finish many other manufacturing techniques have, but this is less of an issue in industrial printers as compared to desktop printers.

1.3 NVBOTS Company Background

NVBOTS, or New Valence Robotics, is a 3D printer startup centered in Boston founded by four MIT students in 2013. NVBOTS hopes to make the entire 3D printer experience very easy for the user. NVBOTS is committed to creating a network of 3D printers that are fully automated, have an easy-to-use interface, and are capable of printing high quality parts. They're known for their printer, the NVPro, and their R&D facility, NVLABS. NVLABS recently developed novel technology that allows for 3D printing multiple metals in the same build. The metals that they're capable to print with include steel, titanium, nickel, aluminum, zirconium, silver, and palladium. Their NVPro thrives in a collaborative environment, like classrooms as NVBOTS has an intense focus on education. Traditional 3D printers lack a queuing system, the STL file must be directly loaded into the AM machine, and the part must be removed manually. The NVPro solves the issue of always needing to be physically at the printer. See appendix A for a peek at the web interface and the older model of the NVPro. NVBOTS lies in between the industrial and desktop realm – a realm that is relatively untapped. Because of the 24/7 automation, courtesy of their cloud based interface (see appendix), the NVPro appeals to both markets. NVBOTS hopes to become the standard 3D printer in office, classroom and R&D spaces.

The NVPro is a material extrusion printer that has a resolution of 100 microns, an accuracy 95 microns, and a build volume of 512 cubic inches [6]. The feature that sets the NVPro apart from other material extrusion printers is that it's fully automated — the NVPro is equipped with a cloud-based web service. The printer has a camera that allows you to stream video to any device you want to monitor the print. It also comes with a queueing system and a robotic arm to remove parts that have been printed. This means a user can print 24/7, from any location, and just using their iPhone. In this thesis I work with the NVPro to calibrate the parameters in the context of materials for higher quality prints.

2. Materials and Methods

2.1 General AM Work Flow

Every 3D printer will have a specialized process to go from 3D CAD model to a prototype. For example, a material extrusion 3D printer operates fundamentally differently than a vat polymerization printer but the general AM work flow can be boiled down to generalized steps. There is a modeling phase, a building phase and a finishing phase. Here is a conceptualized six step process of AM [5]:

1. **CAD.** Computer Aided Design is the first step of any AM machine. This creates a 3D object or surface that the user intends to create.
2. **Conversion from CAD to STL.** Thanks to Chuck Hull STL has become the de facto standard for how AM machines interpret our 3D models. STL describes the closed surface geometry of each layer that motivates the slices made by the 3D printer.
3. **Machine Setup.** This is specific to each AM machine. This step includes uploading the STL file and the interpretation of the STL file by the AM machine. Besides basic upkeep like removal of previous parts or loading filament, each machine has its own calibration settings depending on the material. A web-based interface is part of this step when printing with the NVPro — a visualization is provided which makes the STL interpretation clear. The work in this thesis only uses Quantum3D PLA filament.
4. **Build.** This is the autonomous part of the AM process where the machine uses the given material to make the print layer by layer.
5. **Part Removal.** Here the printed part is removed. With NVBOTS this part is automated — the part is removed and the next job in queue is started.
6. **Post-processing.** Here any differences between the print and the CAD model are fixed. For example, support structures/hairs would be removed or surface finish made smoother.

2.2 Material Properties Tests:

2.2A Differential Scanning Calorimetry:

Differential Scanning Calorimetry (DSC) was used to investigate thermal transitions and properties of different colored PLA which are printed on the NVPro. DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of an empty reference and a sample are compared and measured as a function of temperature. The empty reference and the sample holder for this thesis are both 70 μl aluminum oxide ceramic crucibles. The DSC measures the weight of the sample and gives a curve that plots heat flow versus temperature and these curves can be analyzed to give: melting temperature, crystallization temperature, glass transition temperature, heat capacity, and heat of melting. Here's a DSC profile for PLA:

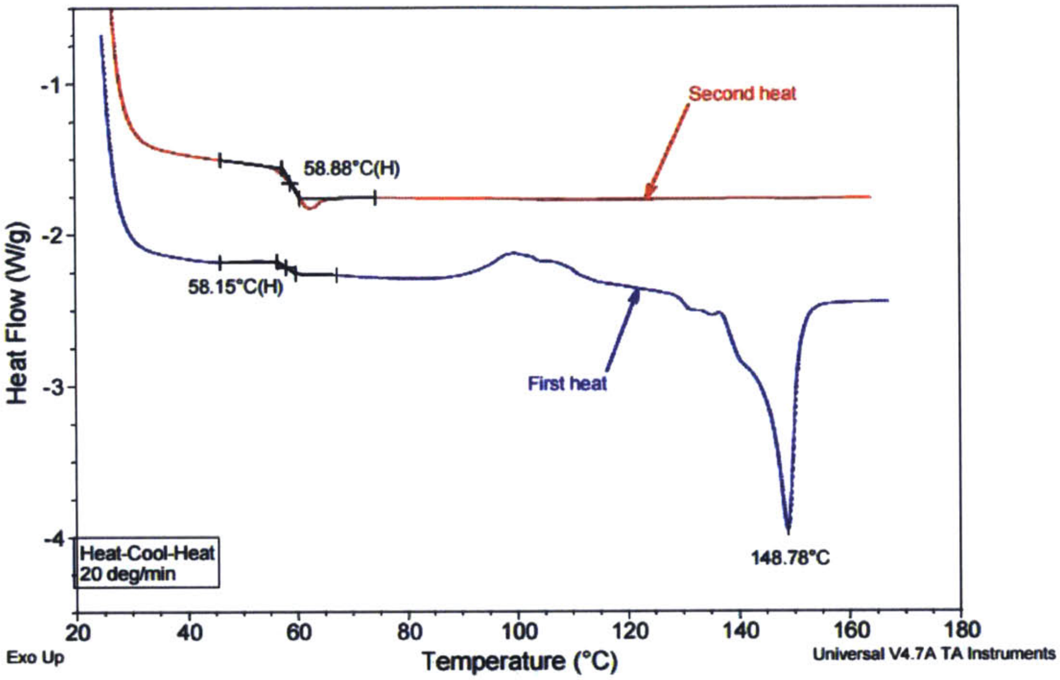


Figure 3. Differential Scanning Calorimetry (DSC) Curves of Processed PLA. This is an example of a DSC running two thermal cycles. You can see how the second thermal cycle gets rid of the thermal history of the processed PLA — the trough at 150C doesn't exist in the second heat cycle [7].

This DSC profile highlights key features of DSC. The glass transition temperature can be seen at around 60C, which is identifiable from the change in slope. The change in slope means the heat capacity of PLA changes once the glass transition temperature is reached. There isn't a peak because no latent heat is absorbed or dissipated — this is because glass transition is a second order transition. The heat of melting can be found by integrating the curve at the melting point which is the temperature that corresponds to the trough — around 150C. For DSC analysis of PLA at NVBOTS, the goal was to investigate the different thermal behaviors of the different colors of PLA and compare the thermal behaviors to the thermal behavior of raw PLA which would give insight on calibrating the NVPro effectively.

2.2B Fourier Transform Infrared Spectroscopy:

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption or emission of a sample. FTIR measures how well a sample absorbs light at each wavelength of light. Each chemical bond absorbs light at different wavelengths and at different intensities so unique chemicals have unique FTIR spectrum. This means that FTIR gives a molecular fingerprint. FTIR is used to identify unknown materials, the quality of a material, and the amount of components in a material [8]. Here's an FTIR spectrum for PLA:

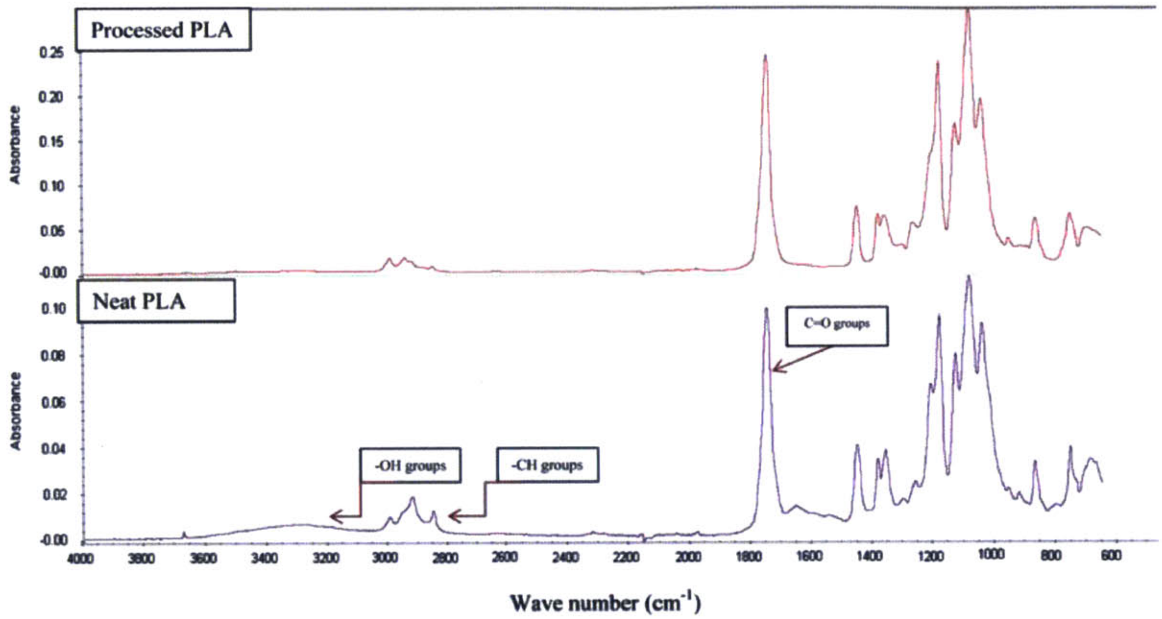


Figure 4. FTIR Spectra for Unprocessed and Processed PLA. Neat is another way of saying the PLA is unprocessed. The FTIR gives a molecular fingerprint where each material has a unique signal. The difference in spectrum here gives insight into the manufacturing process, showing that water is removed during PLA processing [9].

This FTIR spectrum highlights the key features of an FTIR spectrum. FTIR plots descending wave number (a high wave number is high energy) versus absorbance. Neat PLA is raw PLA that hasn't experienced any manufacturing techniques or heat treatments — so PLA pellets rather than the filament used in 3D printing. In the FTIR above, you can see the differences in the spectra in the 2000-3000 cm^{-1} range. These differences can be attributed to stretching vibrations of oxygen-hydrogen bonds — this is likely because water was present in the PLA before it's processed. Water was removed during post processing so you can see water peaks in the neat PLA sample.

For FTIR analysis of PLA at NVBOTS, the goal was to discover the additives in the different colors of PLA and see if these additives have thermal, mechanical or material properties that might give insight on calibrating the NVPro effectively. Investigating the additives and their properties can coincide with the DSC analysis. For example, if the FTIR analysis gives a lot of potential additives but one of the additives has a melting point at 125°C that matches the thermal fluctuations in the DSC at 125°C, it's probably a match.

2.2C Uniaxial Tensile Testing:

Uniaxial tensile testing was used to explore the mechanical property differences between each color of PLA. Understanding mechanical properties on a deep level would allow for intelligent calibration of 3D printers. An Instron, model 4206, was used to see if there was a relationship between color and tensile strength. The tensile test was performed by ASTM standards — ASTM D638 type IV [13]. Here's a CAD model of the dog bone:

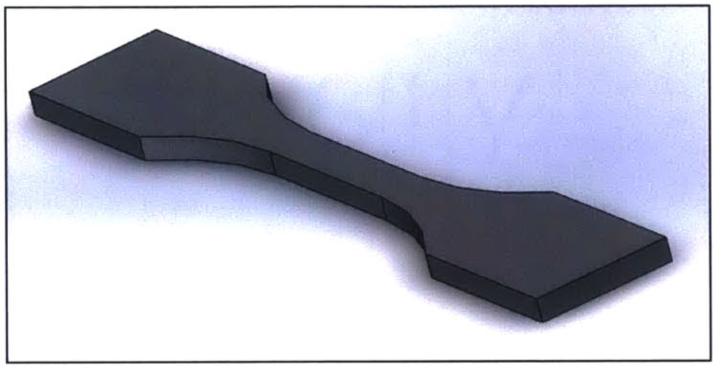


Figure 5. Dog Bone for Uniaxial Tensile Stress. This was printed on the NVPro with an infill percentage of 75% — the maximum infill percentage when working with the online interface. Infill is the percentage of a solid model that should be filled in with material when printed. The length of the narrow body of the dog bone is 33mm.

The instron grabs each head of the dog bone and pulls. Because the body of the bone is significantly thinner than the head the cross-sectional area of the body — using calipers — is used to calculate the stress. All samples failed at loads lower than 1200N at a rate of 5 mm/min. Regardless of whether there's a relationship between mechanical strength and properties like raft peelability, dimensional tolerance or surface finish, characterizing the filament has value.

2.3 Pareto Charts:

Pareto plots are useful when analyzing data about the frequency of problems or causes in a process, when there are many problems but you want to focus on the most significant problem, and when analyzing broad causes by looking at their specific components. In Japanese manufacturing, pareto charts are one of seven basic tools of quality — a designation given to a fixed set of seven graphical techniques identified as being most helpful in troubleshooting issues related to quality [10]. Here's an example of a pareto plot:

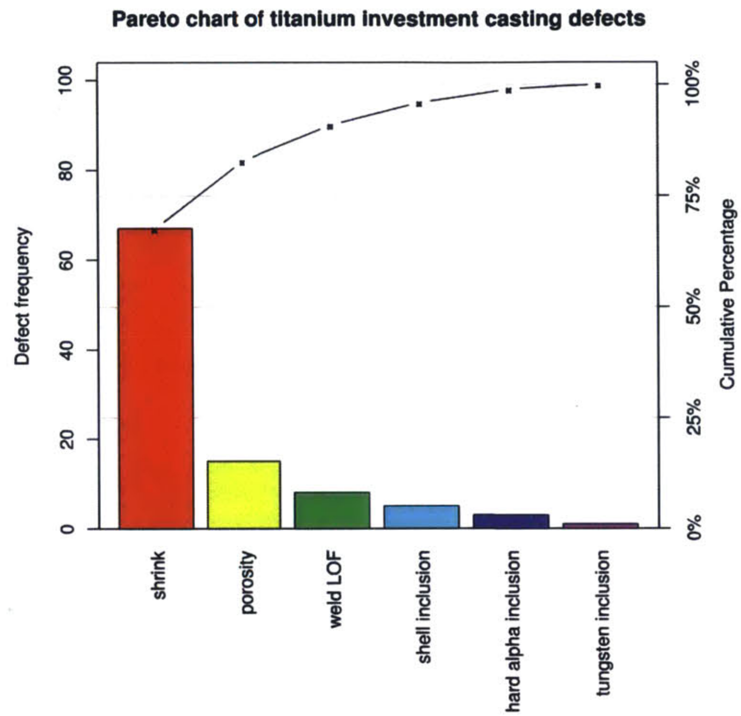


Figure 6. Pareto Plot Example Describing Defects in Titanium Investment Casting Defects. This pareto plot demonstrates that shrinkage is the most dominant problem responsible for defects in titanium casting. Pareto plots are useful in design of experiments — you would solve the shrinkage issue first and foremost since it's the dominant problem [11].

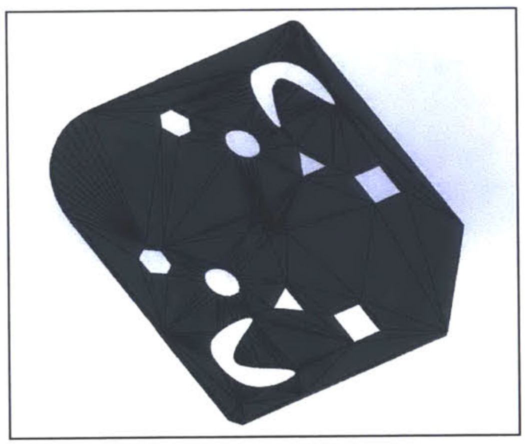
This pareto plot illustrates pareto plots' value. This plot shows us that in titanium investment casting, shrinking is the most important problem to solve and tungsten inclusion is the least important problem to solve. This means solving shrinkage in titanium investment casting has the highest priority — it wouldn't be efficient spending resources or timely/expensive tests investigating tungsten inclusion. For this reason pareto plots are invaluable in design of experiments (DOE). When designing the experiments of this thesis there's a drawback to following the pareto plots because it only accurately assesses the relative importance of the variables if the range is consistent among all of the variables. For example, first layer offset might be the variable that is actually most significant but my scale is off. If I chose a wide temperature range for first layer temperatures but a narrow width range for first layer offset, the pareto plot would incorrectly indicate that first layer temperature is the most significant. However after calibrating, this would be obvious if the samples at the low and high ranges have poor quality. In this thesis, I use the statistical package JMP to construct pareto plots.

2.4 Calibration Tests:

2.4A Alpha Test:

For organizational purposes I refer to different calibration tests as the alpha test, the delta test and the zeta test. The alpha test measures how easily a printed part can be removed from the raft. A Raft is a lattice of filament that is printed before and underneath the intended print. Rafts are primarily used to help with bed adhesion and, particularly key to NVBOTS, to help stabilize print removal. The raft ensures consistent adhesion of the part to the printbed, which is important for maintaining print quality and part removal reliability. By printing the same lattice under every part, only varying in size based on the size of the print, the behavior of the printed part during removal can be predicted and the removal can therefore be reliable. It's easier to wedge the removal blade under a raft then it is a part directly printed to the bed. The larger the part, the harder it is for the blade to get underneath a part if it's printed directly on a bed, rather than on top of a raft. In short, the raft works with mechanism that removes parts automatically and makes it effective reliably. However, separating the print from the raft is something that needs to be fine-tuned. If the raft doesn't separate from the printed piece, this isn't the geometry the user wanted and the broken, half attached raft simply looks bad. The lattice of the raft isn't the kind of resolution anyone wants from their 3D printer. If the raft separates too easily from the first layer, it's usually due to the quality of the first layer being poor, which propagates throughout the print, making the entire print quality poor. So there's a balance — between raft peelability and feature clarity — that needs to be fine-tuned by working with different parameters.

Raft peelability and resolution were balanced by printing a specific part and using a specific scoring system. The scoring system is based on the part below in figure 7:



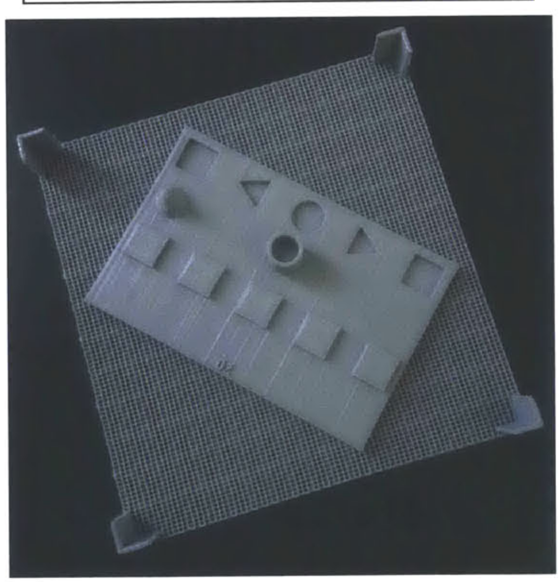


Figure 7. CAD Model of the Alpha Test Part and Raft Example. Figure 7A is the 3D model of the alpha test part and Figure 7B is an example of a raft [17]. The alpha test model is based on making the raft peelable while putting that peelability in the context of what customers care about — quality. A part that peels very easily but has a rough surface or nonuniform surface isn't good enough.

There are 16 features: 2 hexagons, 2 circles, 2 triangles, 2 squares, 2 boomerangs, 2 holes, and 4 corners. Each feature is awarded a half point for having no raft attached after peeling (prioritizing raft peelability) and a half point for having defined features (prioritizing resolution). This means a perfect part will receive a score of 16. The score quantifies the raft peelability and print quality which makes deciding the next calibration steps easier.

2.4B Delta Test:

One of the most obvious trait of a high quality print is dimensional tolerance. Without dimensional tolerance, at best, prints don't look the same as they do in CAD, and at worst, parts warp potentially damaging the printer. Dimensional tolerance was calibrated using a printed part and a set of gauges below in Figure 8:

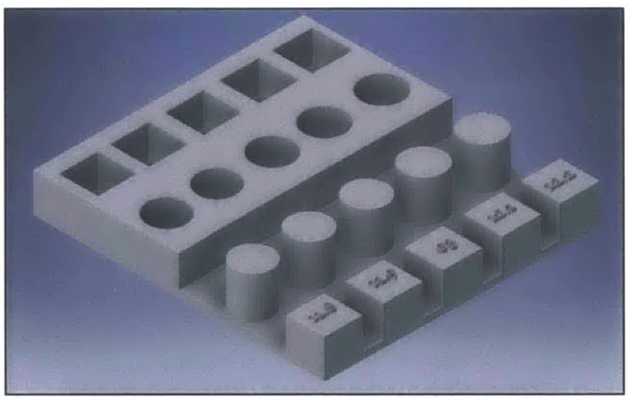


Figure 8. CAD Model of the Delta Test Part and Delta Test Gauges. The cube gauge requires interesting geometry at corners to avoid seams formed during the printing process. Using a coordinate measuring machine (CMM), the female cylinder gauge diameter was 11.983mm with a cylindricity of 0.0348, the female cube gauge had dimensions 12.010mmx12.013mm, the male cylinder gauge had a diameter of 11.901mm, and the male cube gauge had dimensions of 11.984mmx11.973mm.

The scoring system uses a male and female gauge to test male and female features on the printed part. For example, a 12mmx12mm female cube gauge would measure each of the male cube features. If the female cube gauge was too small to get a slip fit on any male features it's awarded 1 point (top of figure 8), if it makes a slip fit on 11.8mm it's awarded 2 points, if it makes a slip fit on 11.9mm it's awarded 3 points, if it makes a slip fit on 12.0mm it's awarded 4 points (perfect – the middle feature), if it makes a slip fit on 12.1mm it's awarded 5 points, if it makes a slip fit on 12.2mm it's awarded 6 points, and if the female gauge was too big to get a slip fit on any male features it's awarded 7 points (bottom of figure 8). This means a perfect part would have a score of 16 and a standard deviation of 0 meaning the two male and two female features have perfect slip fits with the gauges (scores of four for the four features). The score quantifies the dimensional tolerance which makes deciding the next steps for calibration easier.

2.4C Zeta Test:

Another obvious trait of high quality 3D prints is a print with high surface quality. Particularly for fine details, surface finish is the user's first impression of the print. The test for surface quality was done using the CAD model shown below:

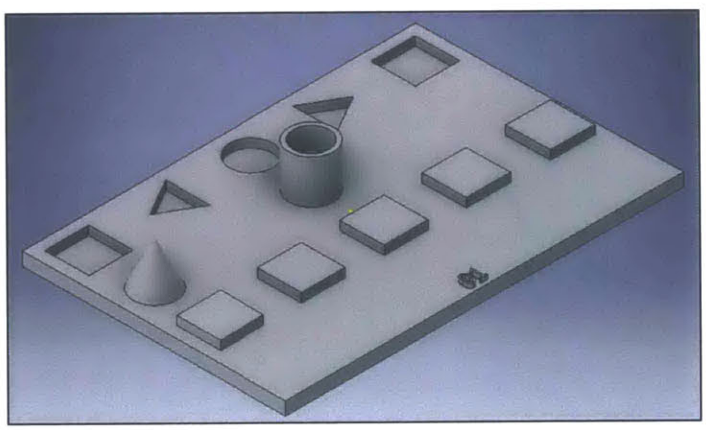


Figure 9. CAD Model of the Zeta Test Part. This was the print used to calibrate surface roughness for each color by varying extrusion multiplier, perimeter acceleration, perimeter speed, and perimeter extrusion width percentage. The balance of the zeta test is in a smooth, clean surface finish and clear, well defined features.

This model has a few types of test features. The cone is a spot where hairs tend to form, which are unwanted side-effects of extruded fiber hair on the feature because the extrusion cannot start or stop without the extra "hair" attached. The half cylinder-half tube feature needs to have a constant radius. The positive and negative squares, triangles, and circles, and the planar areas in between, give the viewer an indication of a standard level of surface roughness. The placement of the features also give different area sizes for testing how the surface finish relates to area size. For example, very small areas are hard to get a nice surface finish on because the machine changes direction too fast, which causes shaking, which causes resonant marks on the surface. The zeta test is scored by ranking the samples — the best surface finish is awarded the highest number and the worst surface finish is awarded the lowest number.

3. Results and Discussion:

This results and discussion is split into two parts: a material properties section and a calibration section. The goal of this thesis is to relate the mechanical, thermal and chemical properties of colored PLA filament to the calibration of a 3D printer for qualities like raft peelability, dimensional tolerance and surface finish for each colored PLA filament. Understanding the relationship between material properties and calibration would allow any 3D printer user to intelligently make their machine print higher quality parts.

3.1 Thermal Testing — Differential Scanning Calorimetry (DSC):

Differential scanning calorimetry tests gave insight on the thermal properties of each of the PLA colored filaments and the raw PLA pellets. A DSC was run from either a temperature range of 25°C to 230°C or a temperature range of 25°C to 200°C. The first run with the DSC had higher temperatures because 230°C is the highest temperature the extruder reaches in the NVPro. For the sake of time, scanning samples to 200°C is faster and less messy especially when doing multiple cycles. At 230°C, the melted PLA strongly adheres to the crucible after solidifying, meaning a solvent is needed in order to remove the PLA from the alumina oxide ceramic crucible. However, since the extruder gets to 230°C, any interesting thermal properties in the 200°C to 230°C should be investigated entirely. The differences in thermal behavior between filament colors from 200°C to 230°C weren't significantly different if you compare figure 10 to figure 12A-G:

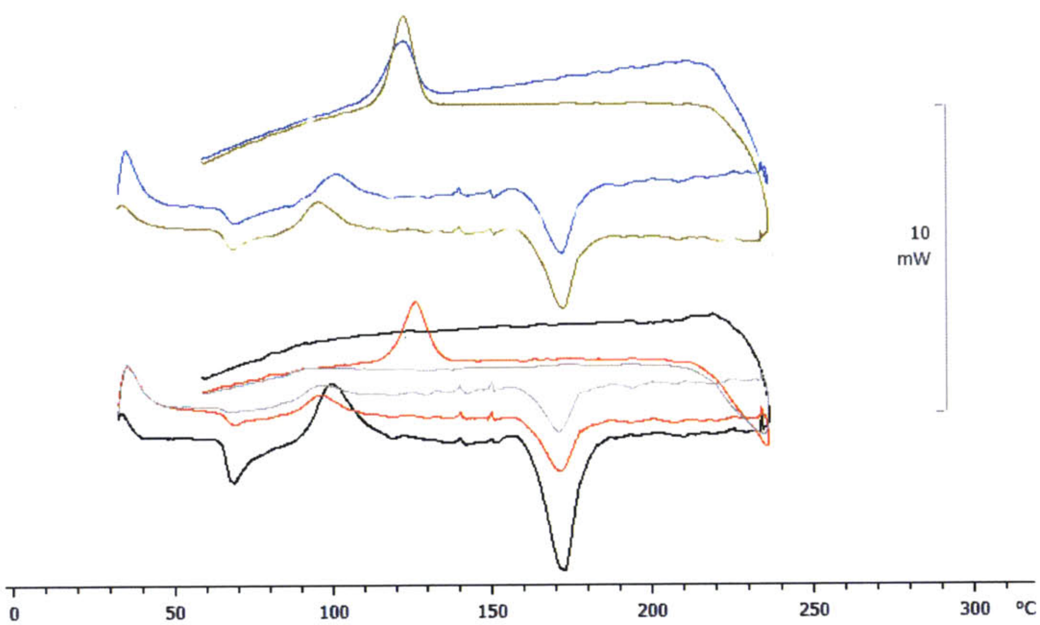


Figure 10. DSC Scan of Colored PLA Filaments from 25°C to 230°C. The placement of these graphs on the Y axis — where Y is the heat flow — is meaningless and just serve as a way to see all the samples on one graph. This means the heat flow of white, grey and red are not lower than the heat flow of blue and yellow. Each color represents the colored PLA — blue, yellow, red and grey — except white is represented by black. Each sample has a unique mass so the relative size of each the peaks aren't comparable.

Looking at figure 10, there aren't many unexplained thermal fluctuations in the 200°C to 230°C range, although red has interesting behavior as it starts to cool down and the heat flow drops faster than the other PLA filaments. There are thermal fluctuations at around 140°C which could indicate an additive in the filament responding to the thermal scan. Another DSC test was needed because the grey and orange (not pictured in figure 10) samples both had interesting crystalline peaks. If you follow the grey curve in figure 10 you can see the small peak at around 100°C — it's hard to accurately integrate a peak that small. In order to get the inherent properties of PLA, the thermal history should be removed by running multiple thermal cycles [16]. Thermal history means there are properties of the material where the material processing — like extrusion — affected the thermal properties rather than being inherent to the material. Erasing the thermal history of PLA is as easy as running two thermal cycles as you can see in figure 11. The second thermal cycle is more representative of PLA because a second thermal cycle gets rid of the processing techniques effect on the thermal data in the DSC curves. Analyzing the DSC results after two cycles weights the inherent properties of PLA more so than the experience of the colored PLA filament as it is being extruded from the NVPro.

After performing three thermal cycles on raw PLA, it was found two cycles were needed to get rid of the thermal history which can be seen in figure 11. The first cycle is arguably the most important because it most closely mimics the process of 3D printing similar to the 230°C scan in figure 10 — the PLA is cut from filament and is heated to 200°C. However, the heating rate is much slower at 10°C/min using the DSC as compared to a 3D printer's extruder in any DSC scan. A 200°C sample placed in ambient temperature will cool faster than 10°C/min. The second cycle is important because it acts as a way to get rid of the thermal history of the PLA filament. Because filament manufacturers keep their manufacturing methods proprietary we don't know, for example, if the heating rates during the extrusion process — from pellets not filament — are equal across all colors. We also don't know what kind of additives are in each color and what kind of effect those additives can have thermally, mechanically or chemically. To determine how many thermal cycles were needed to get rid of the thermal history, raw PLA was cycled three times. The thermal curves for the raw PLA pellet can be seen here below:

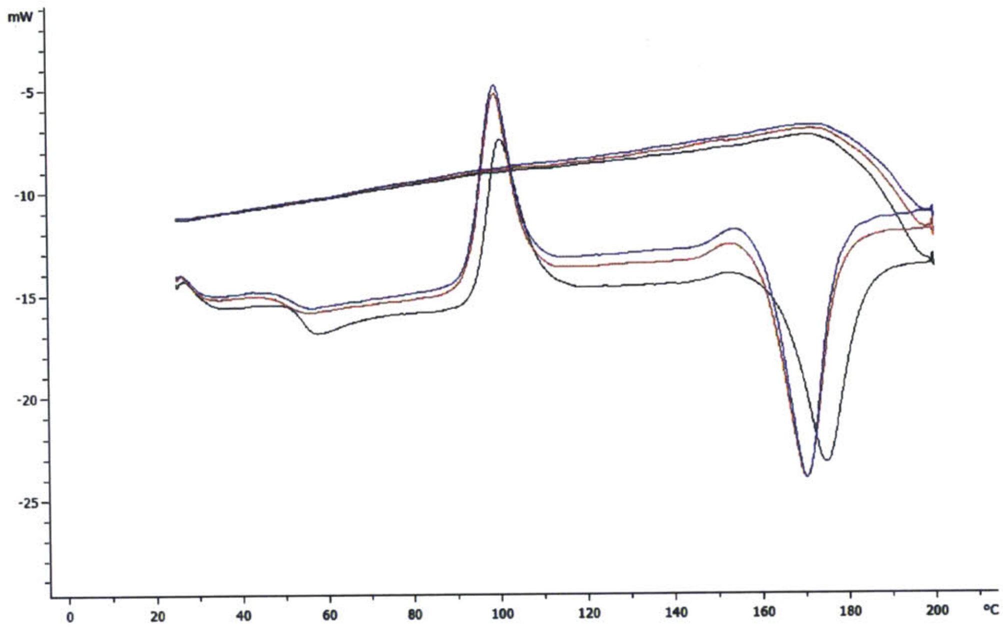


Figure 11. DSC Curves of Three Thermal Cycles of Raw PLA. The first curve is very different while the second and third follow generally the same shape. The melting temperature and crystallization temperature are both higher during the first cycle as compared to the second and third.

Based on the above curve, only two cycles were needed for each color, because the differences in the second and third curve for the raw PLA are negligible. Thermal cycles are important here because polymer processing introduces properties that could not be indicative of our PLA filament. When polymers are extruded — like they must be to yield the rolled filament a consumer buys — the crystallinity of the polymers changes. This is because during extrusion polymer chains are forced to align parallel to the direction of extrusion. As polymers align themselves and the crystallinity gets higher as ordered polymer chains get denser, the strength of the polymers get higher too. In DSC curves, this processing watermark would be obvious because crystalline peaks would be present when in the first cycle but not in the second cycle. However, investigating the first thermal cycle is important to 3D printing because the first thermal cycle tests the thermal properties of the filament — this is the PLA that the 3D printer extruder is directly dealing with. The first thermal cycle is the most important because it is the closest analog to the process going on during 3D printing. However, the second thermal cycle is important because it gives information on the inherent properties of PLA. Investigating the raw PLA pellets has value because every colored filament is made from these pellets and other additives. Comparing the DSC scans of raw PLA to colored PLA allows us to attribute the thermal fluctuations in the colored PLA samples' DSCs to the thermal properties of additives in the colored PLA. The melting temperatures, crystallization temperatures, and glass transition temperatures are similar across the board among colored PLA and raw PLA. The glass transition temperature can be seen around 55°C from the change in slope of the DSC curve without a peak or a trough. The peak indicates the crystallization temperature around 100C and the trough indicates the melting temperature at around 170°C. Each color has a slightly different melting temperature which could be indicative of additives playing with the thermal properties. Here are the DSC curves for each sample:

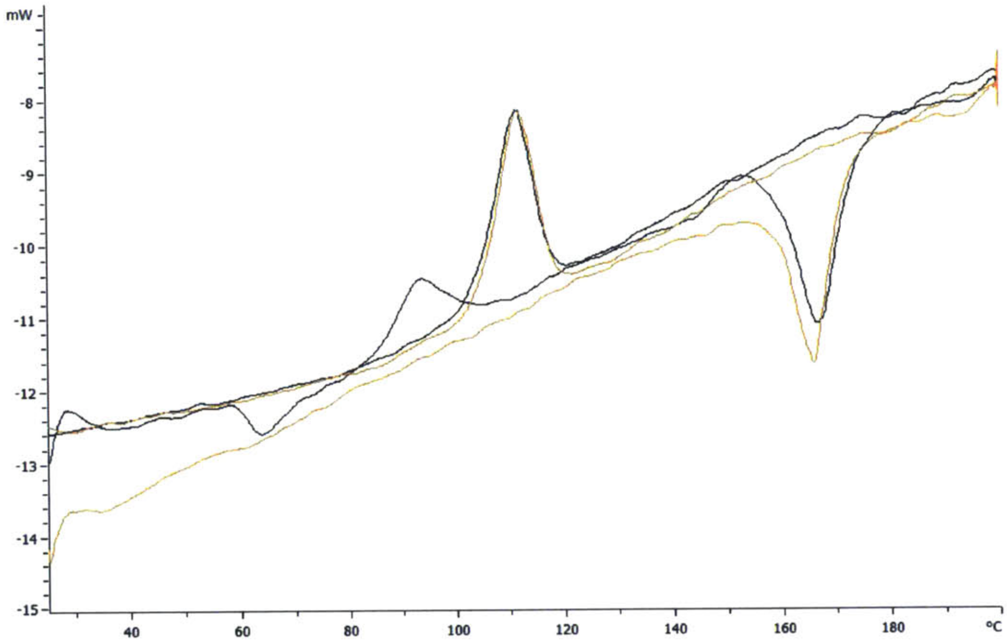


Figure 12A. Two Thermal Cycles of Orange PLA in the DSC. The black curve represents the first cycle of orange PLA in the DSC. You can see the second cycle lacks the crystallization peak present in the first cycle. Additives could be responsible for the thermal fluctuations present between the glass transition and melting temperatures which are not visible in the raw sample.

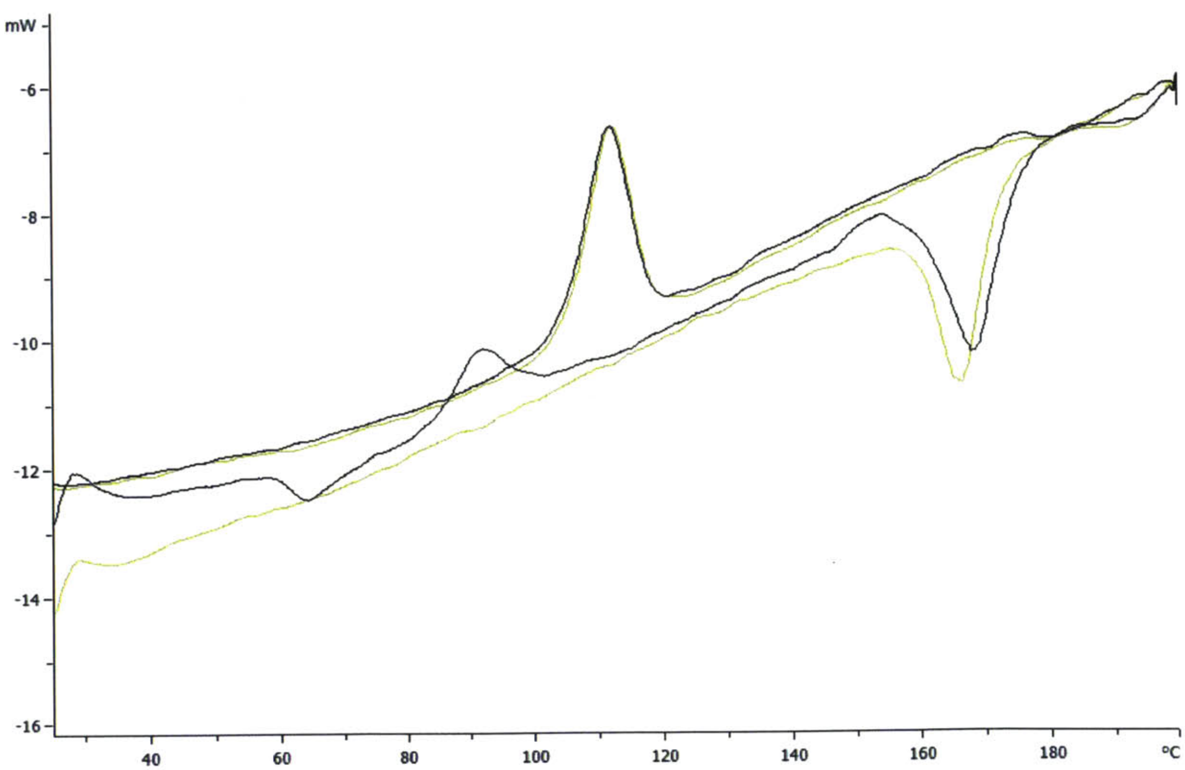


Figure 12B. Two Thermal Cycles of Yellow PLA in the DSC. The black curve represents the first cycle of yellow PLA in the DSC. You can see the second cycle lacks the crystallization peak present in the first cycle.

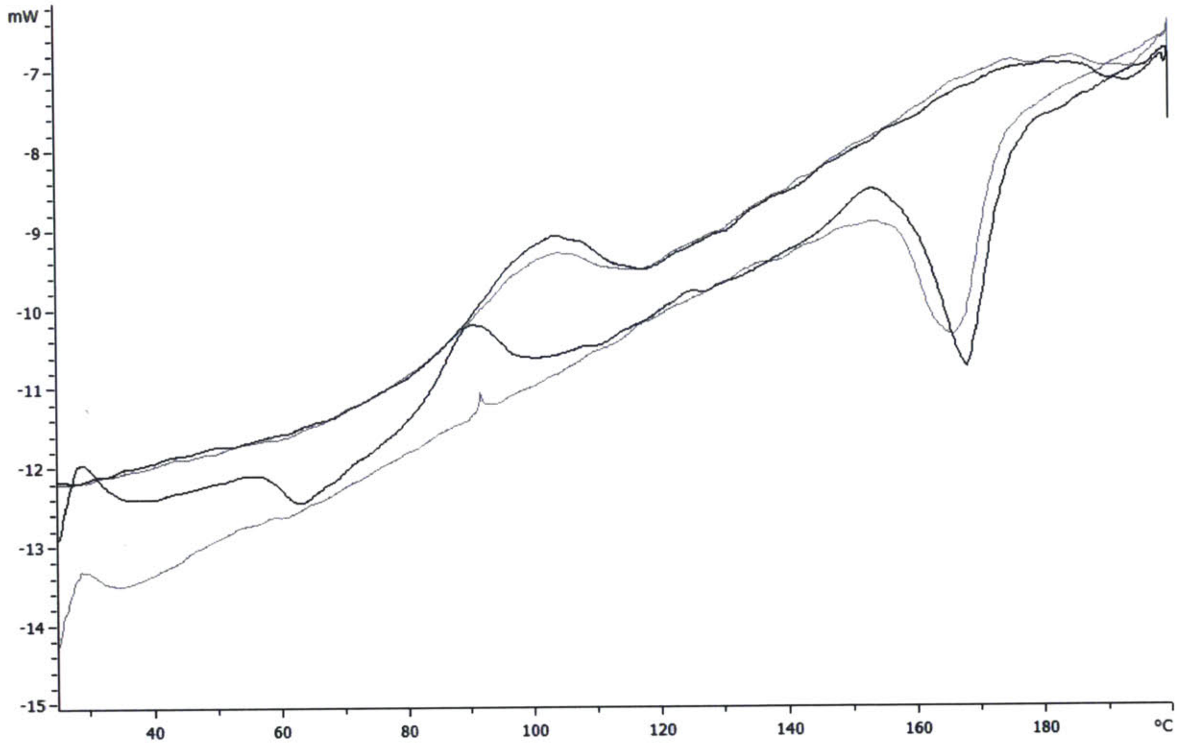


Figure 12C. Two Thermal Cycles of Grey PLA in the DSC. The black curve represents the first cycle of yellow PLA in the DSC. The grey sample is interesting because in the second cycle you can see a slight crystallinity peak.

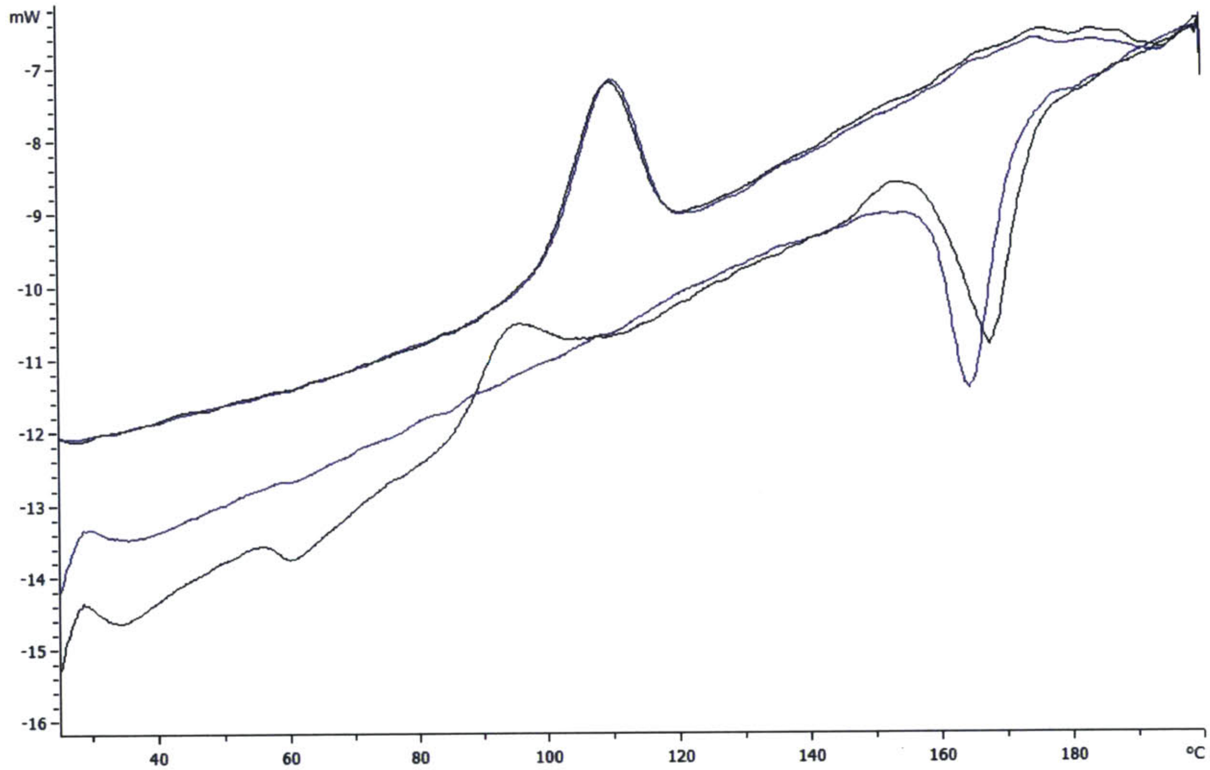


Figure 12D. Two Thermal Cycles of Blue PLA in the DSC. The black curve represents the first cycle of blue PLA in the DSC. You can see the second cycle lacks the crystallization peak present in the first cycle.

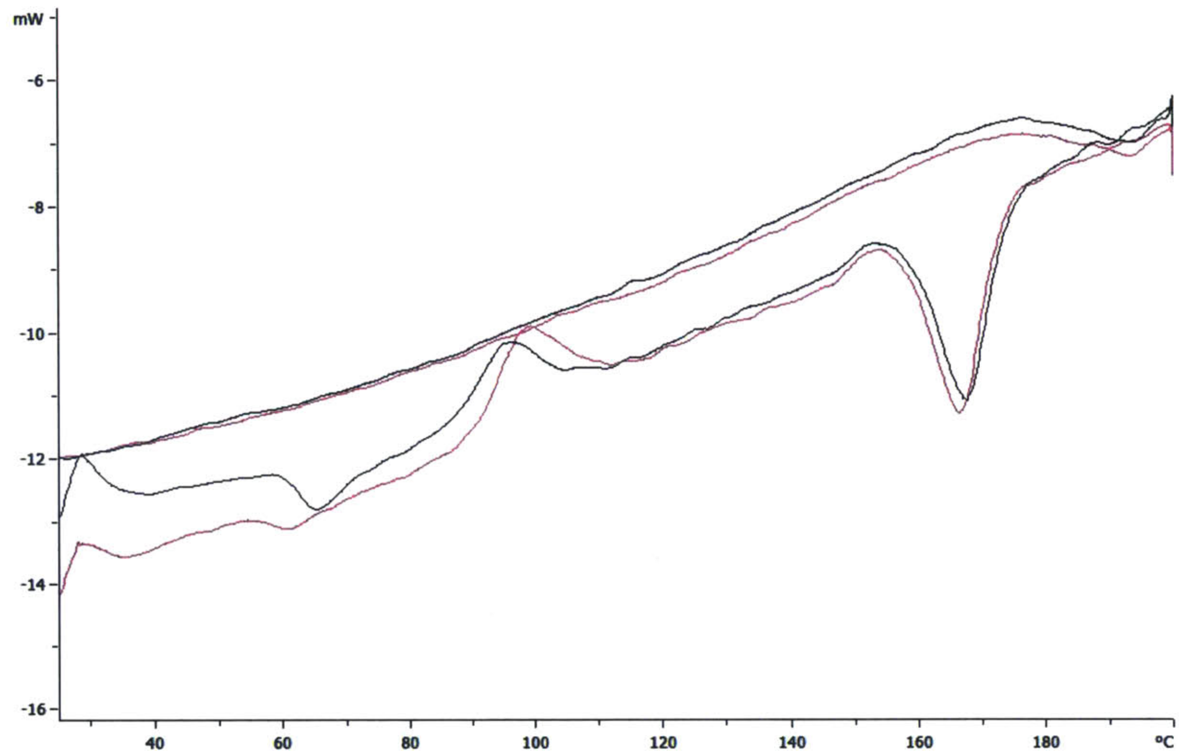


Figure 12E. Two Thermal Cycles of White PLA in the DSC. The black curve represents the first cycle of white PLA in the DSC. You can see the second cycle, represented by pink here, does have the crystallization peak present in the first cycle. Another interesting point is that in either cycles it seems the PLA doesn't solidify which at temperatures as low as 25°C is impossible.

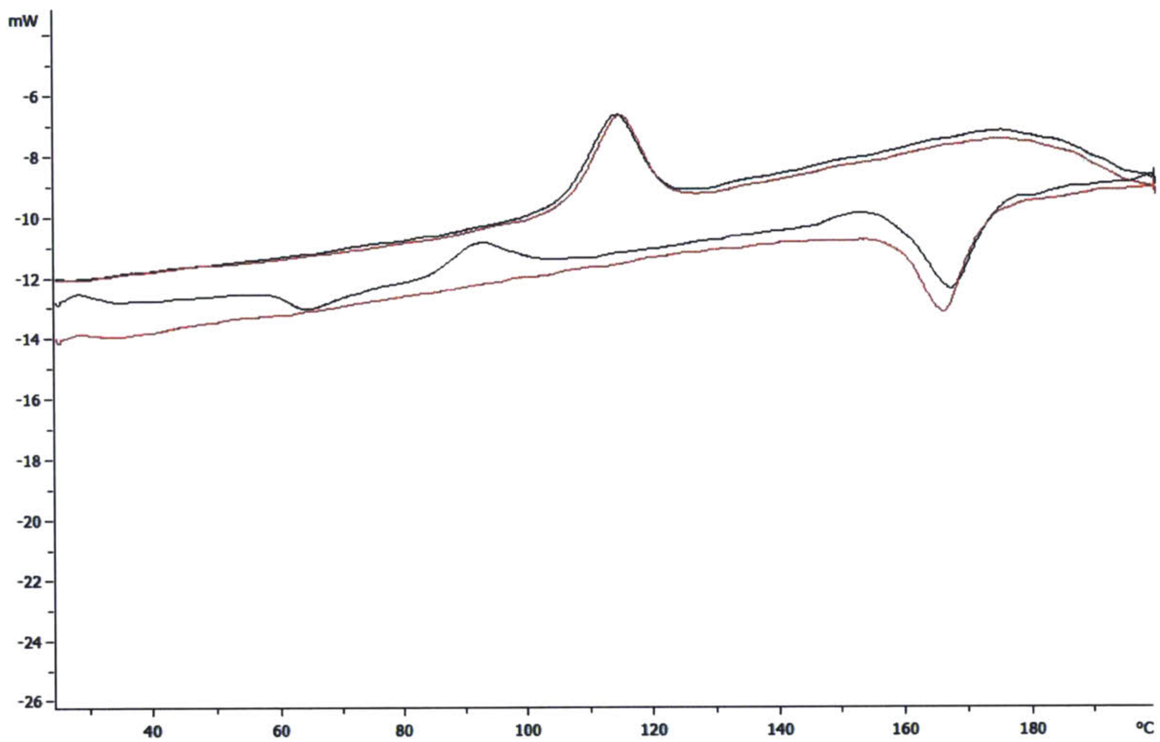


Figure 12F. Two Thermal Cycles of Orange PLA in the DSC. The black curve represents the first cycle of red PLA in the DSC. You can see the second cycle lacks the crystallization peak present in the first cycle.

Each DSC curve has unique characteristics. The slope of these graphs are interesting because they tend to be represented flat. This is most likely an issue of the baseline not subtracting from the samples. However, this is not an issue for calculating the glass transition, melting temperature, the heat of melting, the heat of crystallization, or the degree of crystallization. The thermal DSC data can be summarized in the tables below:

CYCLE ONE: **COLOR**	**HEAT OF MELTING (J/g)**	**HEAT OF CRYSTALLIZATION (J/g)**	**PERCENT CRYSTALLINITY**
Orange	32.9	8.3	12.1%
Yellow	35.6	11.0	12.1%
White	31.0	9.5	10.6%
Blue	29.0	8.3	10.2%
Red	25.4	7.6	8.8%
Grey	22.5	7.5	7.4%
Raw	38.4	24.6	6.8%

Table 1. Thermal Properties of Colored PLA Filament from the First DSC scan. The PLA colors, including a raw pellet of unprocessed PLA, are organized by descending percent crystallinity. The high percent crystallinity of the raw PLA indicates that either additives or the manufacturing process makes the colored PLA filament less crystalline or that the variability in the chemical orientation of PLA makes the colored PLA filament less crystalline.

The raw PLA is less crystalline — more amorphous — than our colored samples. Even at three cycles, the raw PLA still has a crystallinity peak, whereas in general, the colored PLA samples lose this peak after the second cycle. However, the white sample (figure 12E) definitely keeps it crystalline peak and the grey sample (figure 12C) shows evidence of a small peak. During the second thermal cycle the PLA appears to be amorphous because of the missing crystalline peaks. To see the thermal data of the second cycle summarized see appendix B. The PLA filament is more crystalline than raw PLA which indicates that the additives and dyes in the PLA filament or the manufacturing process make PLA filament more crystalline. The previous work with FTIR suggests that manufacturing methods must be playing a role because the strength of signal in the FTIR spectrum didn't completely correlate with percent crystallinity. Filament manufacturers could be flash cooling their extruded filament via an ice or water bath. During extrusion, the temperature of the polymer is over the glass transition temperature which means the polymer chains are free to move around and organize themselves into crystalline domains. However, if cooled too quickly the polymers wouldn't be able to organize themselves into crystalline domains fast enough and the polymer would be more amorphous. Based on our first thermal cycle, the filament is not amorphous on the spool so the cooling rate must not be fast enough to make it amorphous. It's possible that cooling rate would be fast enough to cause the PLA to be amorphous but the additives serve as heterogeneous nucleation sites. Heterogeneous nucleation is when foreign particles or rough surface areas act as primers for crystal to grow. The balance between heterogeneous nucleation and the high cooling rate could be responsible for the increase in percent crystallinity when comparing colored PLA filament to raw PLA pellets. Both PLA types have unique additives and unique processing.

In table 1, the percent crystallinity isn't absolute. I used the latent heat of melting of lactic acid which I would expect to be somewhat similar to PLA. Here's the equation for percent crystallinity:

$$PERCENT\ CRYSTALLINITY = \frac{\Delta Hm - |\Delta Hc|}{\Delta H_m^0} \times 100$$

Where ΔH_m^0 is the latent heat of melting. Although the latent heat of melting of 202.5 J/g for lactic acid [15] isn't going to be the exact latent heat of melting for PLA the percent crystallinity values would scale anyways. Since there are two optical arrangements for lactic acid (L-lactic acid, D-lactic acid), and three optical arrangements for lactide (L-lactide, D-lactide, meso-lactide), there are a variety of PLA structures available. However, since $\Delta T_m°$ decreases with decreasing optical purity — the ability of a chiral molecule to rotate the plane of plane-polarized light — it is expected that using a constant ΔH_m^0 across all optical compositions will introduce error [16]. Basically, the latent heat of melting isn't constant among PLA because PLA varies so much, but it doesn't matter because the relative differences in percent crystallinity is what matters. The percent crystallinity of red for example isn't exactly 8.8% but it's definitely less crystalline than orange. The chemical differences among types of PLA could also be responsible for the crystallinity differences between colored PLA filament and raw PLA pellets.

3.2 Mechanical Testing — Uniaxial Tension:

Mechanical properties are important for more than just understanding PLA and relating it to calibration tests. Since these printers are designed for people to rapid prototype it's important to have a sense for how strong these PLA prints can be. For the engineer, student or designer that needs a robust part, it's valuable to know what magnitude of loads the print can take. Another plus of investigating the mechanical properties is the approachability of mechanical testing. Not everyone that wants to calibrate their 3D printer has access to MIT and its great DSC and FTIR machines. But if mechanical testing can relate to chemical or thermal properties that are hard to test, this type of smart calibration can be more widespread. Mechanical testing for this thesis was done using an instron. The orange PLA dogbone failed at the highest stress and the red PLA dogbone failed at the lowest stress. Table 2 gives a summary of the stress for each PLA color:

COLOR	AVG STRESS ± STD (MPa)	YOUNG'S MODULUS (GPa)	NUMBER OF SAMPLES
Orange	45.1 ± 0.3	1.14 ± 0.01	3
Yellow	41.3 ± 0.5	1.12 ± 0.01	3
Grey	40.8 ± 0.6	1.13 ± 0.01	3
Blue	36.3 ± 0.6	1.14 ± 0.06	3
White	35.6 ± 0	1.13 ± .02	2
Red	32.7 ± 1	0.97 ± 0	2

Table 2. Mechanical Properties of PLA Colors from Uniaxial Tensile Tests. The stress was found by dividing the yield point by the cross section area of the bone body. For the data used to calculate these material properties see appendix C.

The overlaid stress-strain curves for each color can be seen below in figure 13 and individual stress-strain curves by each color can be seen in appendix C:

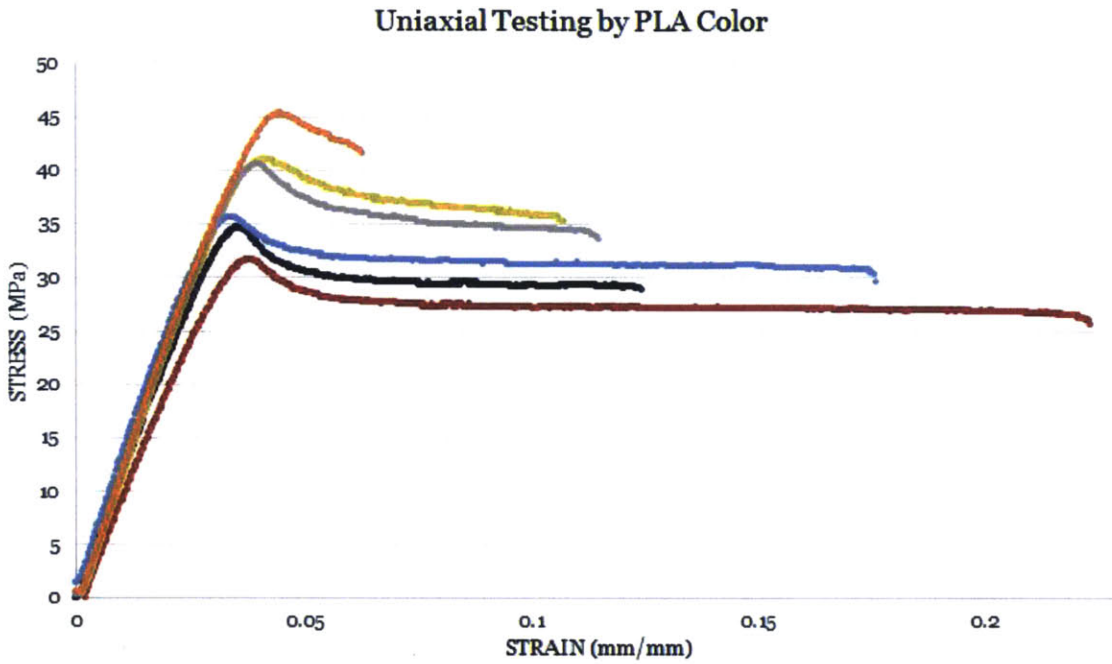


Figure 13. Stress-Strain Curve for the Uniaxial Testing by PLA Color. The yield stress was highest for orange PLA and lowest for red PLA. The extension stops after fracture which you can see with orange PLA around 7% extension.

Looking at figure 13, red PLA can experience up to 3.5 times as much strain as orange PLA and the yield stress of red PLA is 80% of the yield stress of orange PLA. This makes sense considering the percent crystallinity of orange is higher than the percent crystallinity of red. Crystallinity increases yield strength because the secondary bonding is enhanced when the molecular chains are closely packed and parallel. During uniaxial testing when the sample is in tensile stress, the PLA chains orient themselves along the dog bone and become denser. As these polymer chains lengthen and line themselves up next to each other, there are more sites for secondary chemical interactions, these small forces accumulate, and make the sample stronger. This enhanced polymer interchain bonding inhibits polymer interchain mobility – which means we expect a bigger yield stress.

Here's a table showing the relationships between the thermal and mechanical properties:

COLOR	PERCENT CRYSTALLINITY	YIELD STRESS (MPa)	YIELD STRAIN (mm/mm)	YOUNG'S MODULUS (GPa)
Yellow	12.1%	41.3 ± 0.3	0.11	1.14 ± 0.01
Orange	12.1%	45.1 ± 0.5	0.066	1.12 ± 0.01
Grey	7.4%	40.8 ± 0.6	0.12	1.13 ± 0.01
White	10.6%	35.6 ± 0	0.13	1.14 ± 0.06
Blue	10.2%	36.3 ± 0.6	0.18	1.13 ± .02

Red	8.8%	32.7 ± 1	0.23	0.97 ± 0

Table 3. Relationship between Chemical and Mechanical Properties of Colored PLA. There seems to be a proportional relationship between crystallinity and yield stress and an inversely proportional relationship between crystallinity and yield strain. Red PLA also has a significantly smaller Young's Modulus as compared to every other color of PLA.

This table confirms that as crystallinity increases so does the strength of the print which can be explained by polymer chain alignment during uniaxial stress. Young's modulus is a measure of stiffness that should be a material property of PLA — and every color has a comparable Young's Modulus except for red which is clear from figure 13 because the slope is of the elastic region is less than other colored PLA samples. The very high strain of red is likely why the Young's Modulus is low. Future work would include more samples to make these numbers more robust — particularly exploring the Young's Modulus of red more closely.

3.3 Chemical Testing — Fourier Transform Infrared Analysis:

Fourier Transform Infrared analysis gives each material a molecular footprint — each unique material has a unique spectrum. This is useful to us if we can identify the additives then we have insight about how materials will respond to being heated and extruded. The FTIR analysis done on each colored PLA sample, each sample is compared to white because it's expected that white would have the least amount of of additives — considering raw PLA is transparent. Here are the FTIR spectrum for each colored PLA compared to white that confirm this:

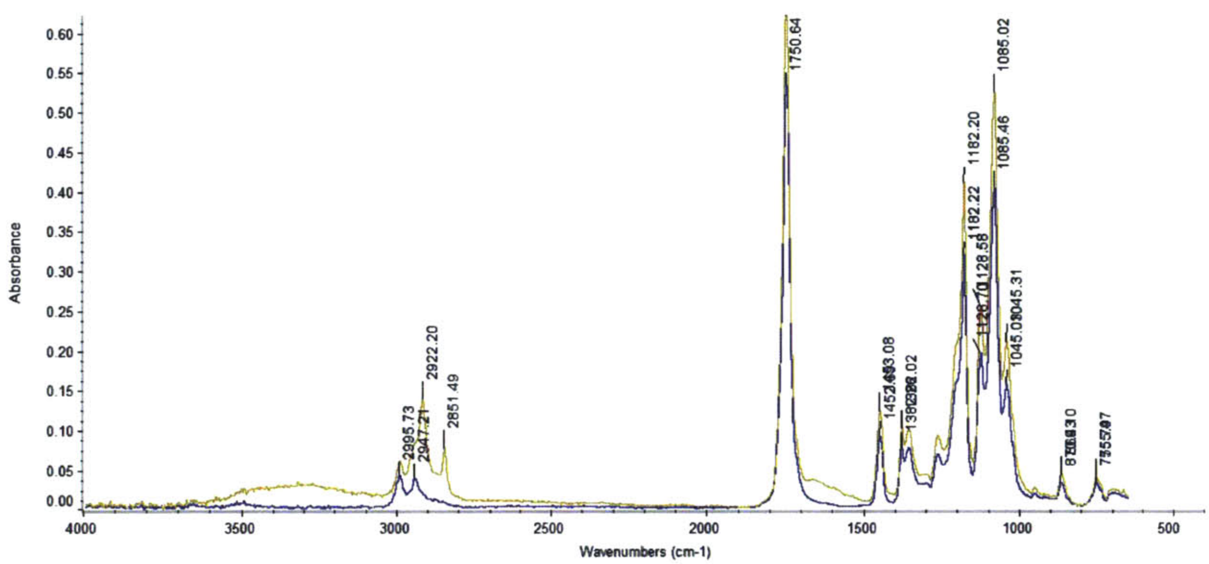


Figure 14A. FTIR Spectrum For White vs Orange. This is the spectrum for both white PLA (represented by blue) and orange PLA (represented by orange). White is expected to have less additives and this is confirmed by looking at the signal near the wavenumber — the x axis — 2900 cm^{-1}.

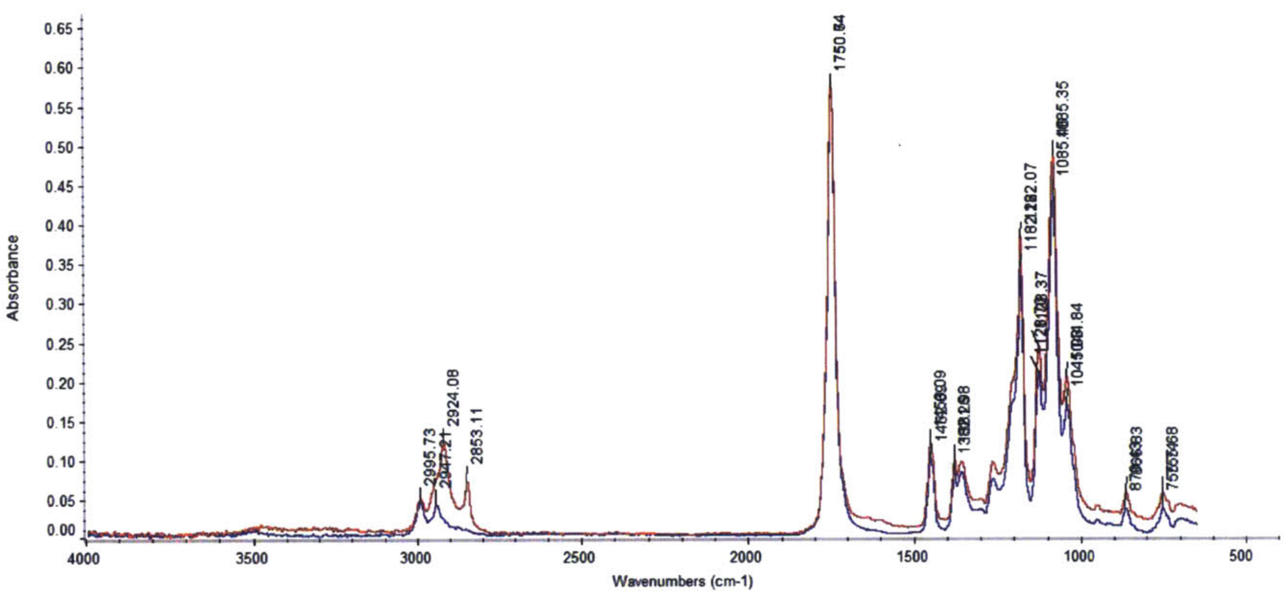


Figure 14B. FTIR Spectrum For White vs Red. This is the spectrum for both white PLA (represented by blue) and red PLA (represented by red). White is expected to have less additives and this is confirmed by looking at the signal near the wavenumber 2900 cm^{-1}.

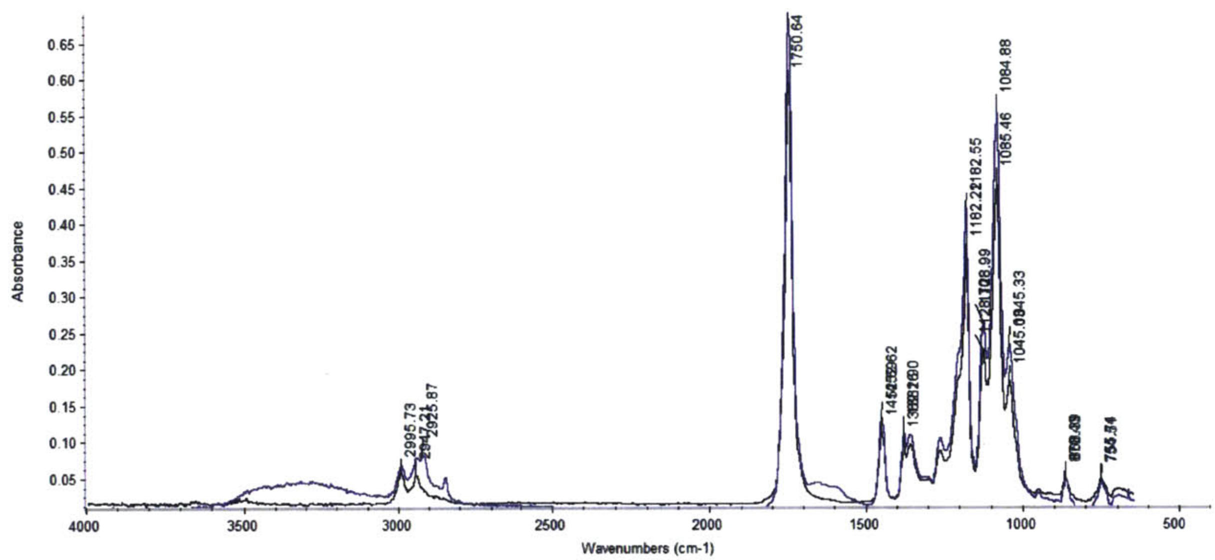


Figure 14C. FTIR Spectrum For White vs Blue. This is the spectrum for both white PLA (represented by black in this case) and blue PLA (represented by blue). White is expected to have less additives and this is confirmed by looking at the signal near the wavenumber 2900 cm^{-1}.

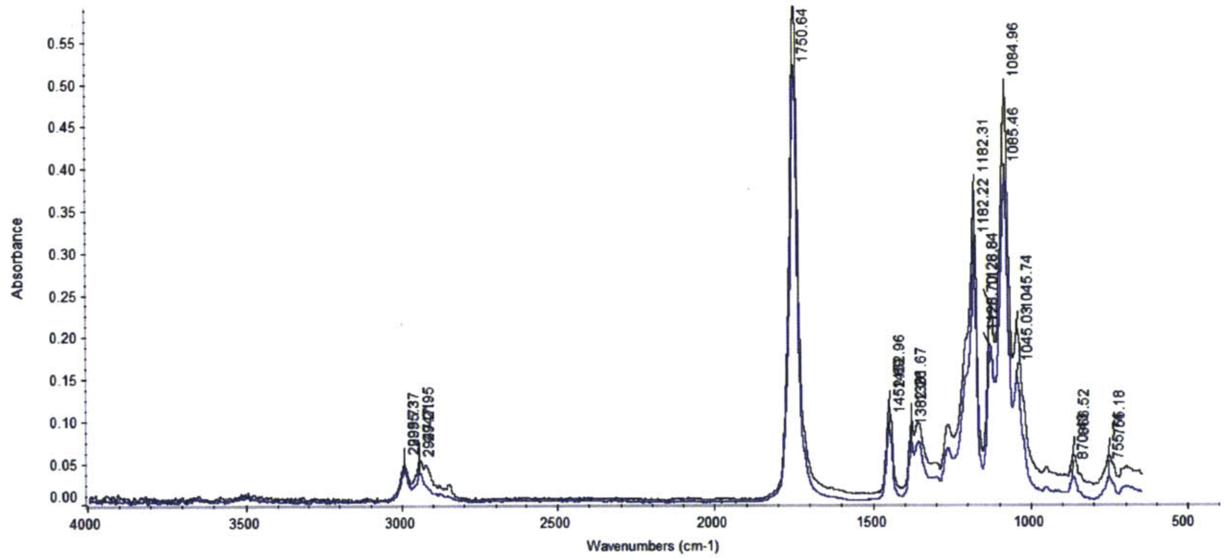


Figure 14D. FTIR Spectrum For White vs Grey. This is the spectrum for both white PLA (represented by blue) and grey PLA (represented by grey). White is expected to have less additives and this is confirmed by looking at the signal near the wavenumber 2900 cm^{-1}.

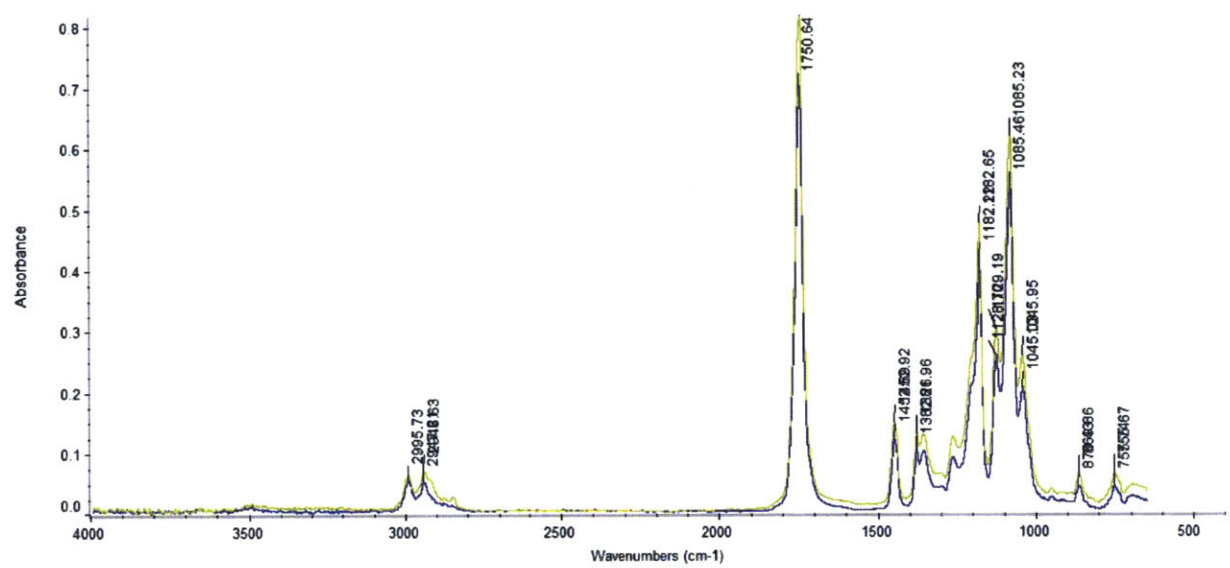


Figure 14E. FTIR Spectrum For White vs Yellow. This is the spectrum for both white PLA (represented by blue) and yellow PLA (represented by yellow). White is expected to have less additives and this is confirmed by looking at the signal near the wavenumber 2900 cm^{-1}.

White has less additives than any of the other PLA colored samples because its spectrum has a smaller signal. White doesn't have the lowest or highest crystallinity so that must mean the chemical additives are more complicated than just plasticizers. If the additives were purely for crystallinity purposes, the DSC data would point to white being the least crystalline. The additives must then play a complicated role in the material properties or could be intertwined with the manufacturing process. It's possible that white is the least crystalline but is cooled slowly during production — making it more crystalline — by the manufacturer Quantum3D. The FTIR libraries were unfortunately not extensive enough to find the exact additives in the colored filaments, but if the additives become known these spectra are useful. Looking at the unique peaks in the spectrum, you can compare the strength of the of the unknown peak's signal to a unknown peak's signal to get concentration of additives. So at the wavenumber of 2900 cm^{-1}, the concentration of the additive can be found by comparing the peak height to a known concentration peak height. The FTIR software predicted that cellulose propionate was an additive — which is a polymer that activated cellulose to polymerize. I don't think the additive is cellulose because the spectra don't match up well, but PLA-cellulose is a common composite for PLA — so it's possible that cellulose is added to commercial PLA filament for added strength.

The chemical properties from FTIR, the mechanical properties from uniaxial testing and the thermal properties from DSC should relate to the calibration tests below. The hope is by studying these material properties, 3D printer users can intelligently decide what settings are best for their machines to get parts with peelable rafts, parts with dimensional accuracy, and parts with a good surface finish. Slic3r, a free 3D slicing software for 3D printers, settings that can be changed with respect to these calibration tests include extrusion multiplier, extruder speed, and extruder acceleration. For example, a polymer that is highly crystalline requires more torque to extrude because a higher crystalline polymer is harder. So a polymer that is very crystalline would probably need a higher extrusion multiplier than a polymer this is less crystalline. The difference between the materials properties investigation and the calibration is that calibration is framed around the materials properties. The material properties are used as a tool to help users calibrate their 3D printers.

3.4 Alpha Calibration Test:

Alpha test is a test of raft peelability — a measure of how well a printed part can be removed from a raft. A Raft is a lattice of filament that is printed before and underneath the intended print. There were five variables tested during the screening design experiment for raft peelability: first layer offset, first layer extrusion width percentage, first layer temperature, first layer speed, and first layer acceleration. As part of the screening design process, a pareto plot made by Paul Burke (see appendix D) found that first layer offset and first layer extrusion width (FLEW) percentage were the most important problems that needed to be tackled. The pareto plot was made by printing a minimum amount of combinations determined by the statistical package software to give us an idea of how each of the settings interact with each other. It determines the biggest problems and found offset and FLEW percentage to be the biggest problems. Since these two variables are the biggest problems we printed many samples changing these two settings between three different values to find what gave us the highest quality print. The highest quality part is quantified by using the scoring system described in figure 7. The variables for the first round of the alpha test can be found in table 4 and the scores can be found in table 5:

ROUND1 SETTINGS	OFFSET (mm)	FIRST LAYER EXTRUSION WIDTH PERCENTAGE
HIGH	0.3	190
MED	0.2	180
LOW	0.1	170

Table 4. List of Settings and their Values for Alpha Test for Orange. The offset, or the distance between the lines in the first layer, had a value of 0.1 mm, 0.2 mm, and 0.3 mm. The first layer extrusion width percentage, or the extrusion multiplier of the first layer, had a value of 170%, 180% and 190%.

PART ID	OFFSET	FLEW	FULL	PARTIAL	TOTAL SCORE
1	H	H	8	8	12
2	M	H	5	11	10.5
3	L	H	0	4	2
4	H	M	6	10	11
5	M	M	11	5	13.5
6	L	M	0	4	2
7	H	L	6	10	11
8	M	L	8	8	12
9	L	L	0	4	2

Table 5. Scores for the Alpha Test for Orange. The optimal first layer offset and extrusion width were found to be 0.2 and 180 respectively. This means that the first layer lines traced out by the 3D printer should be spaced out by 0.20 mm and extrude 180% more material.

For data on other colored filament besides orange see appendix E. From the pareto plot in appendix D, the most important variable is the first layer extrusion width percentage.

COLOR	EXTRUSION WIDTH PERCENTAGE	PERCENT CRYSTALLINITY	YIELD STRESS (MPa)
Red	182%	8.8%	32.7 ± 1
Orange	180%	12.1%	45.1 ± 0.5
Grey	174%	7.4%	40.8 ± 0.6
Yellow	172%	12.1%	41.3 ± 0.3
White	172%	10.6%	35.6 ± 0.6
Blue	170%	10.2%	32.7 ± 1

Table 6. No Clear Proportional Relationships between First Layer Extrusion Width Percentage, Percent Crystallinity, and Yield Stress. Red and Blue have the lowest percent crystallinity and yield stress but have respectively the highest and lowest extrusion width percentage. For permutations including the variable offset see appendix E.

Although there isn't a simple positive linear relationship between first layer extrusion width percentage and crystallinity that doesn't mean there aren't any relationships at all. This assumes that the scoring system is perfect and relates entirely to the quality of the part which is too optimistic. A white print potentially won't get a score of 12, 13 or 14 which means that although all six colors have comparable max scores the scores shouldn't be treated equal. The white filament struggles to achieve these high scores as consistently as orange and yellow do. For example, the average score for white PLA during the first round of the alpha test was 5.7 whereas the average scores for orange and yellow during the first round of the alpha test was 7.6 and 7.7 respectively. Each PLA color is not perfectly calibrated for the NVPro — isn't likely to be be able to be boiled down to one variable or calibration by color wouldn't be a problem worth investigating.

The PLA colors that seem to have the best raft peelability are orange and yellow while the color that struggles with raft peelability the most is white. Self-adhesion is stronger as crystallinity goes up in chlorinated isotactic polypropylene [17] so it's expected that PLA would follow suit as a fellow semi-crystalline polymer. A polymer that self-adheres is going to have a harder time peeling from a raft. The percent crystallinity of orange and yellow is higher than the percent crystallinity of white so it would be reasonable to expect orange and yellow to have worse raft peelability. Additionally, it's important to take in account the heat flow rates of each PLA sample. Here is a table of white, yellow and orange heat flow rates:

COLOR	HEAT FLOW RATE (~W/°C)	AVG FIRST ROUND SCORE
Yellow	0.041	7.7
Orange	0.028	7.6
White	0.023	5.7

Table 7. Heat Flow Rates and Average First Round Score for Orange, Yellow and White PLA. Yellow and orange peel from the raft easily because the cooling rate is high — cooling faster means the sample is less crystalline and self-adhesion is less significant.

The heat flow rate was found by taking the slope of the DSC curves as they begin to cool from 200°C. The change in heat flow is different among different colors of PLA filament. Cooling liquid semicrystalline polymers very quickly makes them more amorphous and cooling liquid semicrystalline polymers very slowly makes them more crystalline. Orange filament and yellow filament tend to peel off the raft significantly easier than white filament. This can be explained by the heat flow rates in table 7. The yellow PLA is cooling nearly twice as fast as the white PLA. A scoring system that ensures all PLA filament colors are calibrated equally well is needed to expand this concept to all colors. Figure 14A-14E do look like they have more of a slope than traditional DSC curves which explains why these slope values are fairly close to zero.

3.5 Delta Calibration Test:

There were five variables tested during the screening design experiment for dimensional tolerance — a measure of how accurately the NVPro will print a length CADed to be a particular length. If my STL says the NVPro should print a 10 mm by 10 mm by 10 mm cube but the z direction is off by 2 mm then it has poor dimensional tolerance. The variables used in the initial test were: extrusion multiplier, perimeter acceleration, perimeter speed, and perimeter extrusion width percentage. The screening design process found that external extrusion width and perimeter speed were the most important variables. A screening design process is immensely helpful because 3D prints take time. Assuming I want a low, medium, and high setting for each of these four variables, that would mean I would need to budget time for 3^4 or 81 prints. It takes about 3 hours for a delta test part to print so that's around 240 hours. However, with a fractional factorial design process we can cut the number of prints down to a low 18 — just 60 hours.

The delta test was a test for calibrating dimensional tolerance — testing to see how closely a length CADed to be length Xmm is to the printed length. A pareto plot was designed in order to determine which settings have the biggest impact on dimensional tolerance. The variables tested were: extrusion multiplier, perimeter acceleration, perimeter speed, and perimeter extrusion width percentage. The extrusion multiplier controls the extrusion flow rate, and is given as a factor, e.g. 1 means 100% and 1.5 means 150%; perimeter acceleration is the acceleration of the extruder during the perimeters, so the outer layer of the print; and perimeter speed is the speed of the extruder; and perimeter extrusion width percentage controls the extrusion flow rate on the perimeter. The data behind the scoring used here can be seen in appendix F and the process is described in section 2.3 of materials and methods. The pareto plots, organized by each settings of low, medium and high value, can be seen here:

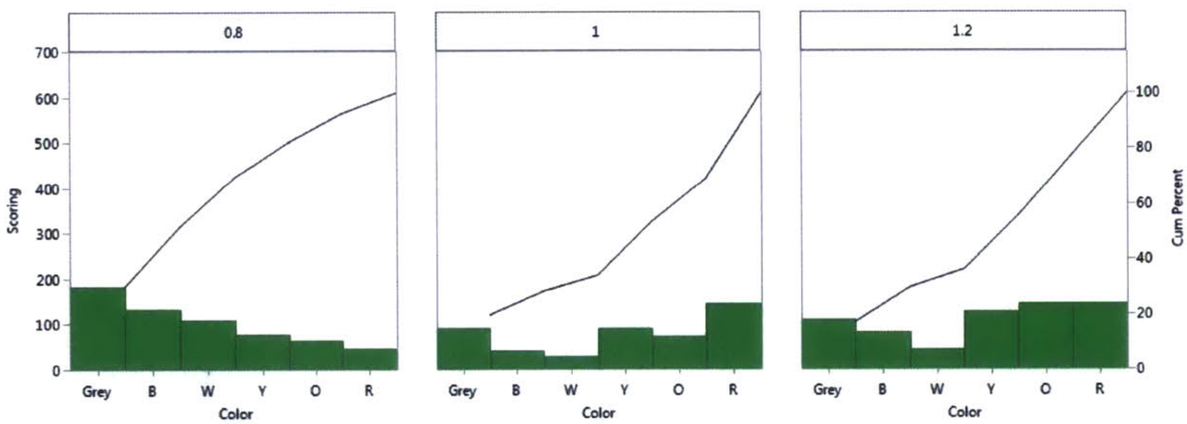


Figure 15A. Pareto Plot for Extrusion Multiplier. Each box represents E multiplier values of 0.8, 1 and 1.2 and has an x axis where B represents blue, W represents white, Y represents yellow, O represents orange, and R represents red. The scores for each color are moderately different for the same settings which means extrusion multiplier is moderately dependent on color.

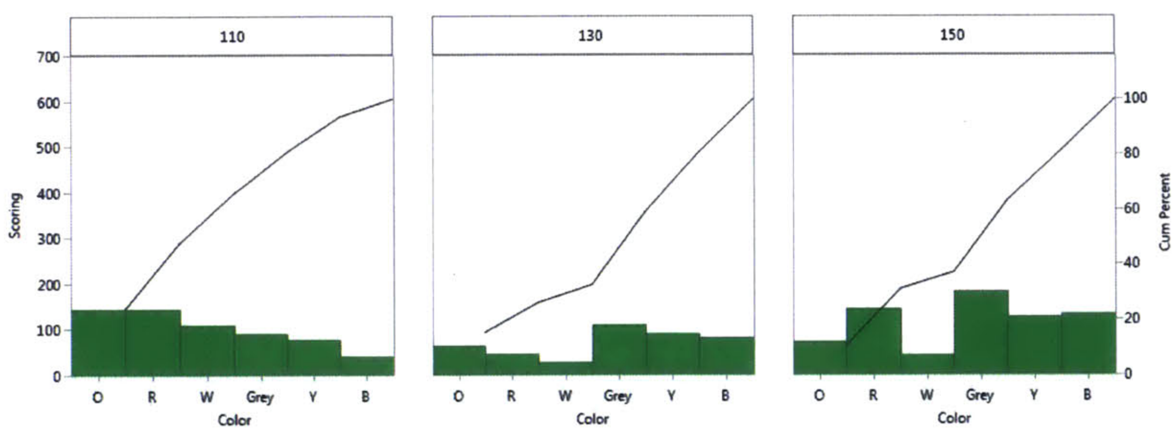


Figure 15B. Pareto Plot for External Perimeter Extrusion Width (EPEW) Percentage. Each box represents EPEW percentage values of 110%, 130%, and 150%. The scores for each color are different under the same settings, particularly speeds of 150%, which means EPEW is dependent on color.

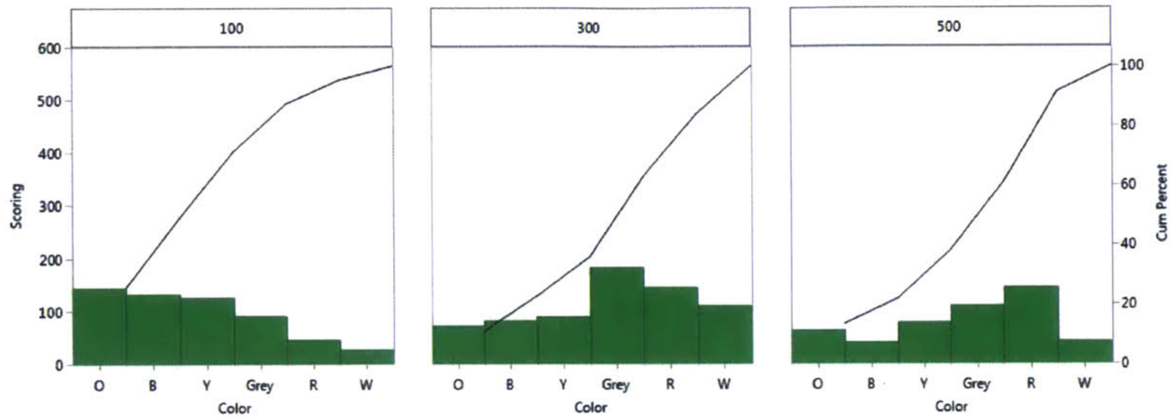


Figure 15C. Pareto Plot for Perimeter Acceleration. Each box represents accelerations of 100 m/s^2, 300 m/s^2, and 500 m/s^2. The scores for each color are moderately different for the same perimeter acceleration which means perimeter acceleration is moderately dependent on color.

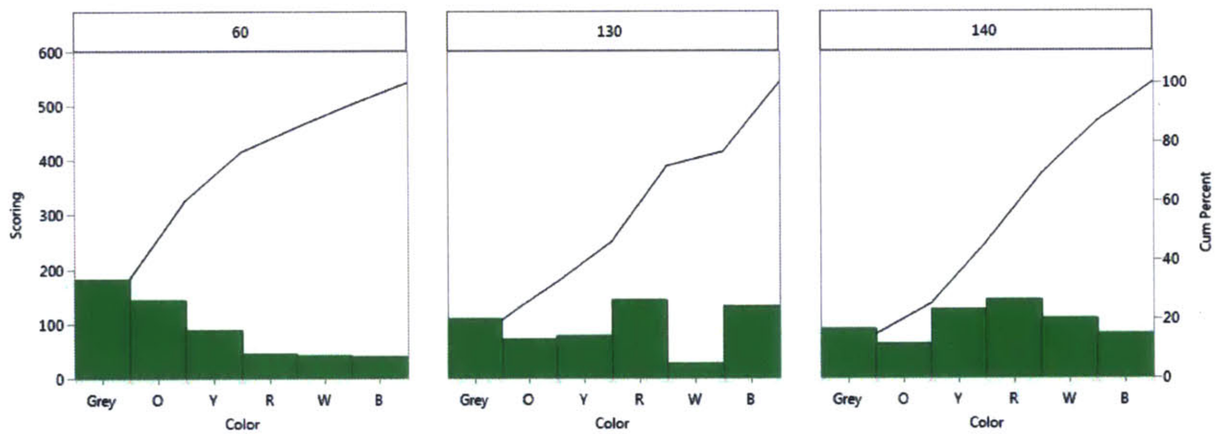


Figure 15D. Pareto Plot for Perimeter Speed. Each box represents perimeter speeds of 60 mm/s, 130 mm/s, and 140 mm/s. The scores for each color are significantly different for the same perimeter speed which means perimeter speed is dependent on color.

All of these pareto plots are scaled based on the score — a measure of the relative quality. To determine which variables are the most statistically significant, you look at the graphs by variable and choose based on how uneven the bars are across the color axis of the plot. Based on these pareto plots it seems the most quality dependent variables are EPEW percentage and perimeter speed. However, the EPEW percentage values are high relative to the calibration done in the Zeta calibration which could potentially mean the pareto plot is inflating the importance of the EPEW percentage. The calibration process is outlined in alpha and the same method would be used for dimensional tolerance but with a different scoring system and different settings — EPEW percentage and perimeter speed. I would expect there to be a relationship between crystallinity and the EPEW percentage of the dimensional tolerance. As crystallinity increases the torque required to extrude goes up because the polymer chains cannot align themselves as easily because of the limited mobility of interchain secondary bonding. The detail in the features of this calibration test are dependent on how much PLA is extruded. For example, a male feature that has been over-extruded will have a larger feature size than what the STL says it should be and would receive a lower score. Since higher crystalline materials require more torque to extrude it makes sense that a high crystalline material would be under-extruded under the same settings as a low crystalline material. To balance this, an increase in the EPEW percentage is needed. Along those line, a lower crystalline material would need a lower EPEW percentage because it over-extrudes. This is also balanced out by perimeter speed. Extruding equal volumes of PLA slowly across the perimeter increases the amount of PLA extruded as compared to a fast perimeter speed.

3.6 Zeta Calibration Test:

There were five variables tested during the screening design experiment for surface roughness: extrusion multiplier, perimeter acceleration, perimeter speed, and perimeter extrusion width percentage. Because of time issues, I was able only to do multiple rounds with orange and red. I chose orange and red because they have very different percent crystallinity and qualitatively orange PLA gives higher quality prints and red PLA gives lower quality prints. The first round data for all PLA colors can be found in appendix G. The first and second rounds for red and orange are in tables 8-13 below:

ROUND1 VARIABLES	EXTERNAL-PERIMETER EXTRUSION-WIDTH PERCENTAGE	PERIMETER SPEED
HIGH	1.05	130
MED	1	120
LOW	0.95	110

Table 8. Settings for the Round One Zeta Test for Red and Orange PLA. The external perimeter extrusion width (EPEW) percentage controls the amount of material extruded for the outermost layer of the 3D printed part. The perimeter speed controls the speed of the extruder as it prints the outermost layer of the 3D printed part.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	H	L	1
2	M	M	2
3	L	H	5
4	M	L	6
5	L	M	4
6	H	H	3

Table 9. Orange PLA Scores for the Round One Zeta Test. L, M and H correspond to the settings values in table 8. A score of 6 means the highest quality print of the set and a score 1 means the lowest quality print of the set.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	H	L	5
2	M	M	1
3	L	H	4
4	M	L	2

5	L	M	3
6	H	H	6

Table 10. Red PLA Scores for the Round One Zeta Test. L, M and H correspond to the settings values in table 8. A score of 6 means the highest quality print of the set and a score 1 means the lowest quality print of the set.

ROUND2 VARIABLES	EXTERNAL PERIMETER EXTRUSION WIDTH PERCENTAGE	PERIMETER SPEED (mm/s)
HIGH	1	110
MED	0.95	100
LOW	0.9	90

Table 11. List of Settings for the Round Two Zeta Test for Red and Orange PLA. The external perimeter extrusion width (EPEW) percentage controls the amount of material extruded for the outermost layer of the 3D printed part. The perimeter speed controls the speed of the extruder as it prints the outermost layer of the 3D printed part.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	H	L	1
2	M	M	6
3	L	H	2
4	M	L	4
5	L	M	3
6	H	H	5

Table 12. Orange PLA Scores for the Round Two Zeta Test. L, M and H correspond to the settings values in table 11. A score of 6 means the highest quality print of the set and a score 1 means the lowest quality print of the set.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	H	L	1
2	M	M	6
3	L	H	4
4	M	L	5
5	L	M	3
6	H	H	2

Table 13. Red PLA Scores for the Round Two Zeta Test. L, M and H correspond to the settings values in table 11. A score of 6 means the highest quality print of the set and a score 1 means the lowest quality print of the set.

Based on calibration data from the NVBOTS engineering team, the most important setting for surface finish is external perimeter extrusion width (EPEW) percentage and perimeter speed. Therefore Zeta calibration doesn't have an accompanying pareto plot like the Alpha and Delta tests. Red PLA calibrated in the second round had an EPEW percentage of 95% and a speed perimeter of 100 mm/s, and orange PLA in the second round calibrated had an EPEW percentage of 95% and a perimeter 90 mm/s. These calibrated values make sense in the context of the material properties of orange and red PLA. The EPEW percentage and perimeter speed are very related to the flow rate of extruded material. High EPEW percentage and low perimeter speed both result in more extruded material. During extrusion, the temperature of the polymer is over the glass transition temperature which means the polymer chains are free to move around and organize themselves into crystalline domains. When being extruded, polymers that are highly crystalline require more torque to extrude because polymer chains have to align and densify in order to go through the aperture of the extruder. Both calibrated red and orange PLA had EPEW percentage settings of 95%. Orange PLA filament has a higher percent crystallinity (12.1%) and a calibrated perimeter speed of 90 mm/s and red PLA filament has a lower percent crystallinity (8.8%) and a calibrated speed of 100 mm/s. At an equal EPEW percentage to red, Orange PLA filament requires a lower perimeter speed because it is more crystalline and needs the extra push to extrude an equal amount of PLA.

4. Conclusion & Future Work:

3D printing is a hot topic in manufacturing and a truly useful tool, but it has limitations. Print quality properties — like raft peelability, dimensional tolerance and surface roughness — are hard to calibrate perfectly. Calibrating can theoretically be explained by material properties but it's not always simple. There are steps 3D printer users or manufacturers can take to make their colored PLA prints better. A solution likely more accessible to major manufacturers — like Quantum3D — could be annealing. Filament manufacturers should anneal their spools post extrusion to increase the percent crystallinity of colors like red and blue (8.8% respectively 10.2%) to the percent crystallinity of colors like yellow and orange (12.1% respectively 12.1%). Having relatively the same percent crystallinity in each color would mean that many of the material properties would be similar as well. Crystallinity related to thermal, mechanical and chemical testing done in this thesis. Annealing a filament spool would require heating the spool between the glass transition temperatures and the melting temperature of PLA (between 40°C-50°C and 160°C-180°C), allowing the polymer chains to flow freely and organize themselves into crystal domains. If the cooling is controlled during this time, the percent crystallinity could be the same across all colors of PLA filament. If cooled more slowly, the percent crystallinity will get higher since this allows time for the polymer chains to arrange themselves in ordered lamellar domains. If all PLA colored filaments had the same crystallinity, the similar crystallinity across color would mean many properties would be similar across color as well. Printing different colored PLA prints all under the same settings will not give you very different levels of quality parts.

The more average user can calibrate the settings on their 3D printer with respect to each color. Orange and yellow PLA filament should have their extrusion multiplier settings increased because of their high percent crystallinity — with the NVPro there was success in lowering the perimeter speed which meant more extruded PLA. The extruder temperature for the first layer — the layer printed after the raft — was also turned up from 190C to 195C and 198C for red PLA and white PLA respectively.

For future work, more samples would give more confidence to the data. The FTIR analysis was only one spectrum for each color, rather than having an average of spectra. The uniaxial testing and the DSC were both more robust and had multiple runs for averages, or for determining trends. For chemical testing, I would run a raw piece of PLA as well. Comparing the spectrum across colors is an effective way to illustrate the subtle differences in additives, but it would be interesting to see the effect of PLA's organic chemistry as you compare an FTIR spectrum of the PLA pellets to a spectrum found in polymer libraries. For thermal testing, there are two additional runs that I would do. First, I would run a DSC on colored PLA that has been printed — so cutting a small piece off a print. It would be interesting to see if the DSC curve of this printed colored PLA matched the DSC curve for the second thermal cycle. I'd expect the curves to not be the same. Although the polymer is reheated during extrusion, the extrusion process would undo any of the annealing done during this cycle. I'd also like to buy commercial raw PLA filament. It would be interesting to compare the DSC curves of raw PLA pellets to raw PLA filament to see the effects of the manufacturing process or to see if manufacturers add the same additives manufacturers do in colored PLA filaments, but just not dyes. Raw PLA had the lowest crystallinity so it would be interesting to find out if the raw PLA filament would have additives that increased its crystallinity to be on order with the colored filament or if the percent crystallinity would just go up from extrusion before going on a spool. Subtracting the raw PLA filament spectrum from any colored PLA spectrum would leave only the signal responsible for dyes. For the delta test, I would like to implement the calibration process done during the alpha test to see if there's a relationship between dimensional accuracy and the amount of material extruded. The amount of material could be a function of the crystallinity of the colored PLA, the external perimeter extrusion width (EPEW) percentage, and/or the perimeter speed. For the zeta test, I would like to develop the STL further so there are more features that would define a high quality surface finish. I would also like to incorporate more settings because EPEW percentage and perimeter speed tend to control the surface quality of features better than the surface quality as a whole — like large planar areas of a print. Lastly, I would like to anneal spools before they are put on the 3D printer to see if that can control print quality — specifically annealing and cooling slowly PLA filament like red which aren't as crystalline. If these get explored, having the capability to produce high quality 3D printed parts reliably — one of additive manufacturing's biggest weaknesses — could be a lot more accessible.

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6. Appendices:

6.1 Appendix A:

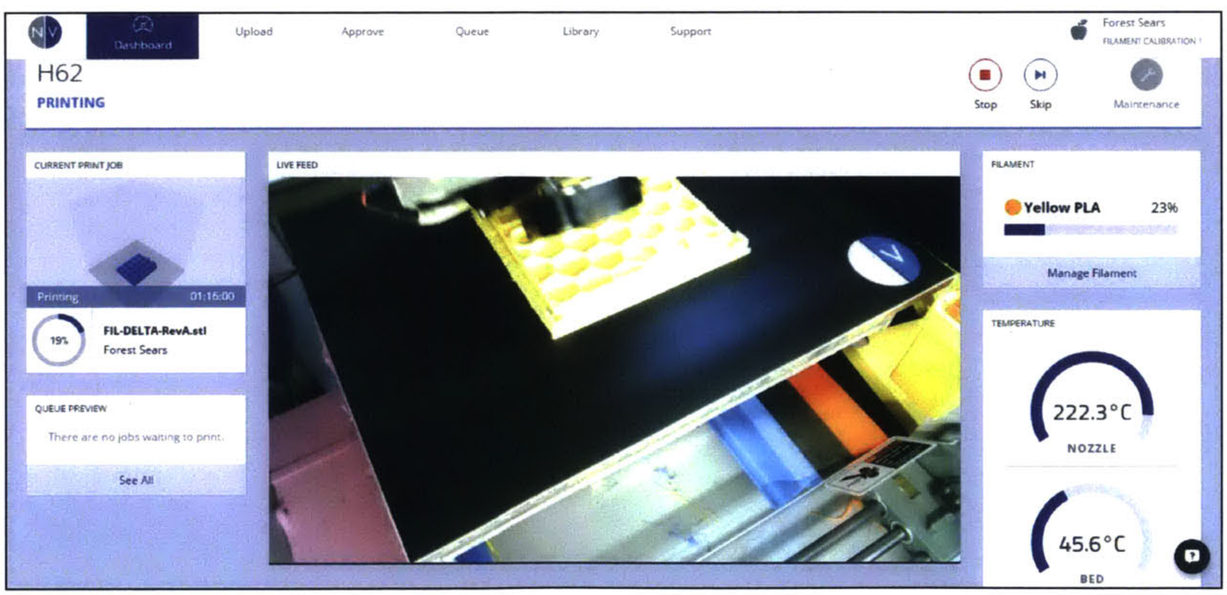

Figure A. Screenshot of the web interface.

6.2 Appendix B:

Here is a summarized table of the thermal DSC data for the second thermal cycle:

CYCLE TWO: COLOR	HEAT OF MELTING (J/g)	HEAT OF CRYSTALLIZATION (J/g)	PERCENT CRYSTALLINITY
Raw	-42.8	26.4	8.1%
White	-30.8	14.1	8.2%
Yellow	-29.6	0	—
Blue	-28.2	0	—
Orange	-27.9	0	—
Grey	-27.1	0.3	13.2%
Red	-24.0	0	—

Table B. Thermal Properties of PLA color from the second DSC scan. The PLA colors and raw PLA are descending by percent crystallinity. The yellow, blue, orange, and red samples all had no crystallinity peaks which means that the polymer is now amorphous instead of crystalline.

6.3 Appendix C:

Here are stress-strain curves for each PLA color which were used in part to find the values in table 2:

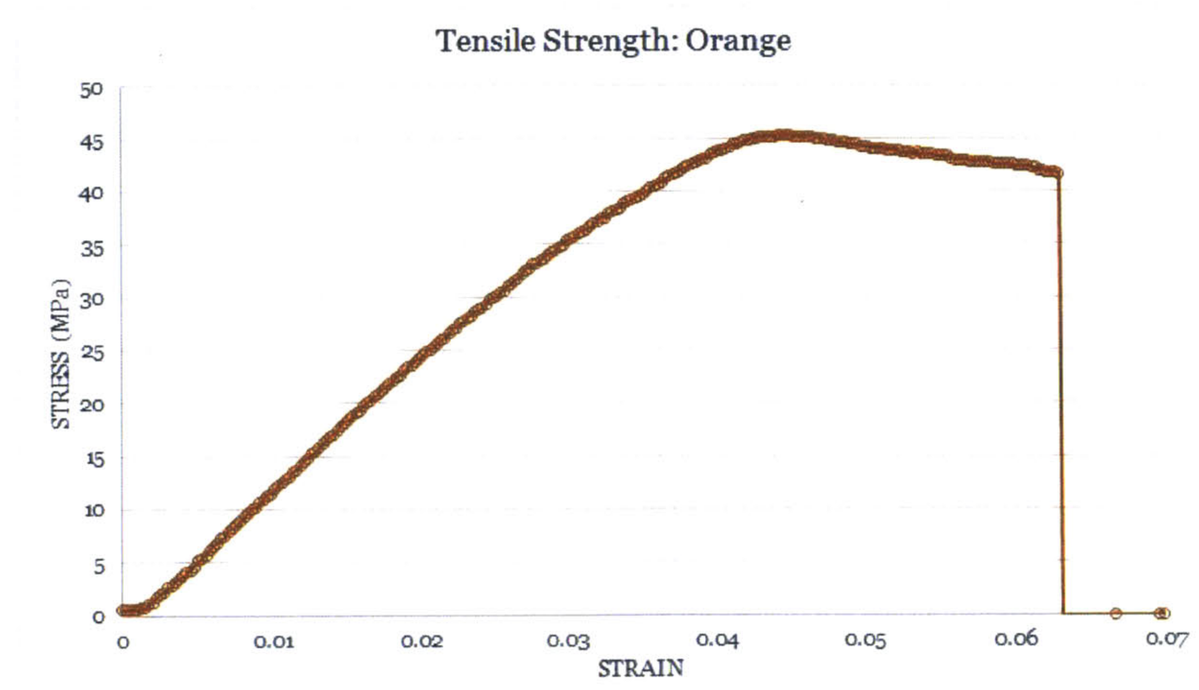

Figure C-A. Tensile Stress-Strain Curve for Orange PLA. The stress, in MPa, is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain, which is dimensionless, was found by dividing the extension by the body length of the dog bone — 33mm. Orange had the highest yield stress at 45.1 ± 0.3 MPa.

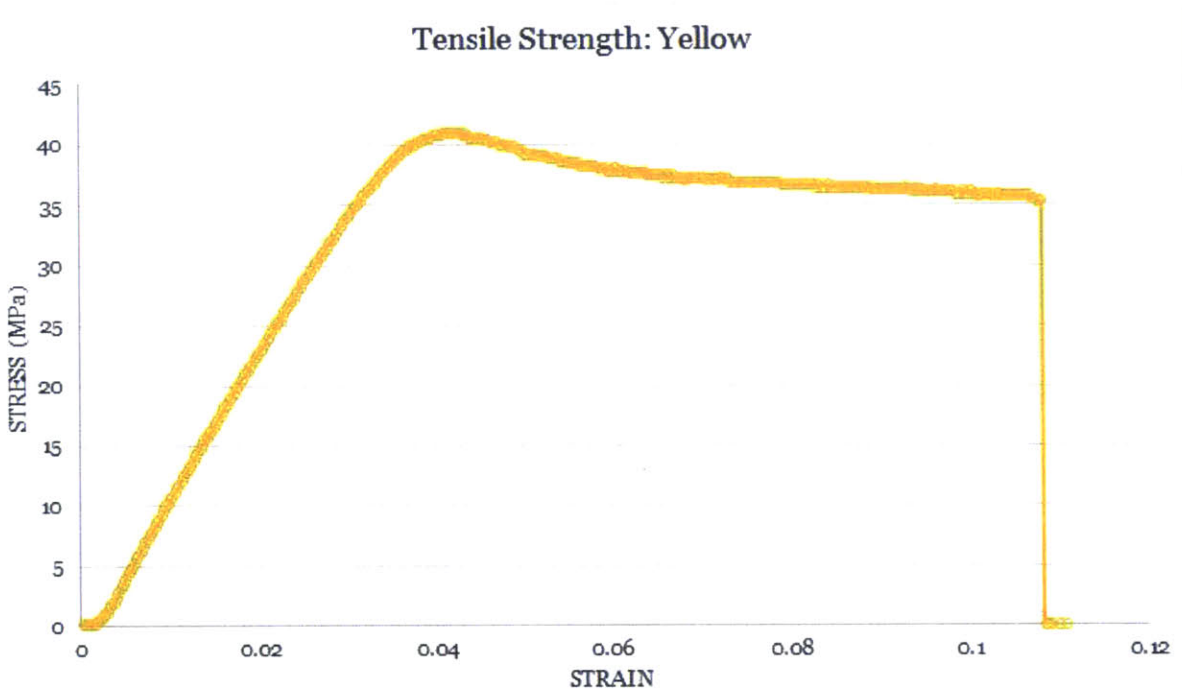

Figure C-B. Tensile Stress-Strain Curve for Yellow PLA. The stress, in MPa, is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain, which is dimensionless, was found by dividing the extension by the body length of the dog bone — 33mm.

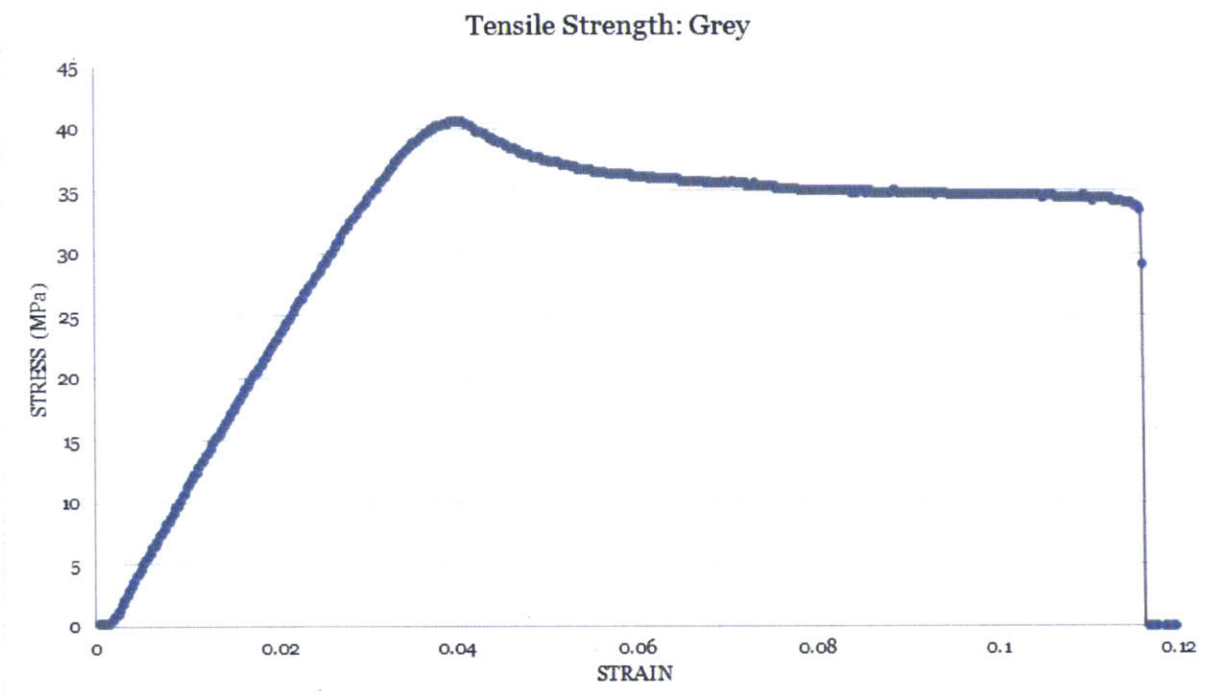

Figure C-C. Tensile Stress-Strain Curve for Grey PLA. The stress, in MPa, is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain, which is dimensionless, was found by dividing the extension by the body length of the dog bone — 33mm.

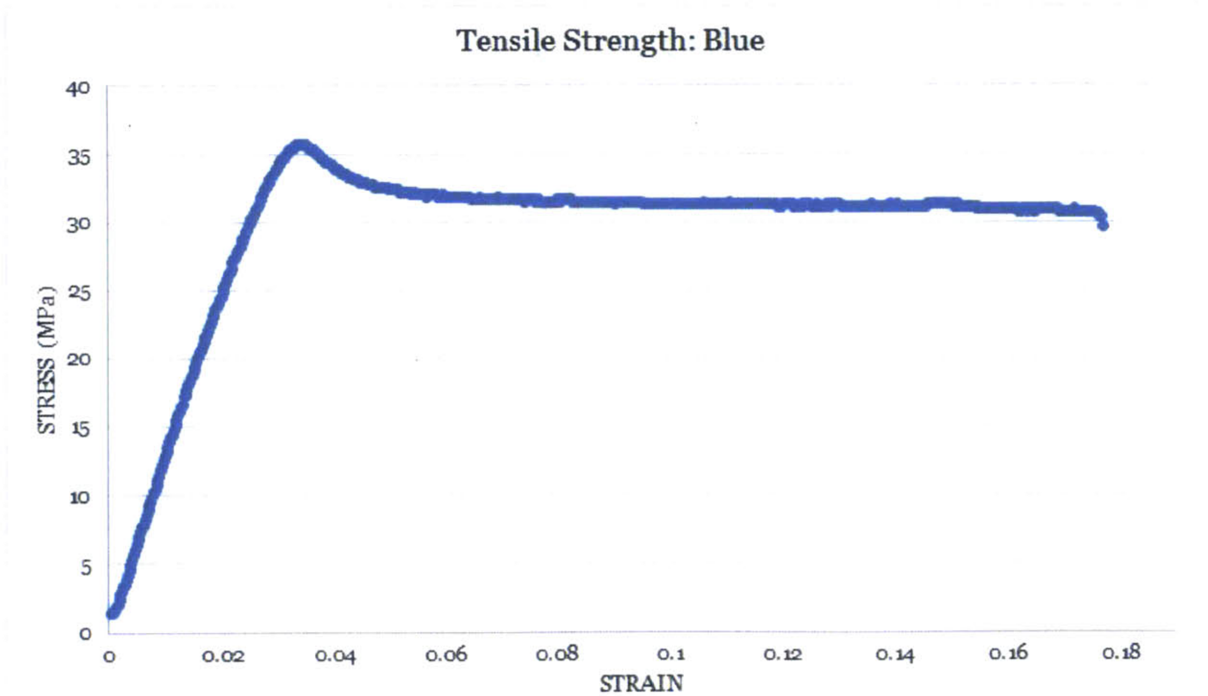

Figure C-D. Tensile Stress-Strain Curve for Blue PLA. The stress, in MPa, is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain, which is dimensionless, was found by dividing the extension by the body length of the dog bone — 33mm.

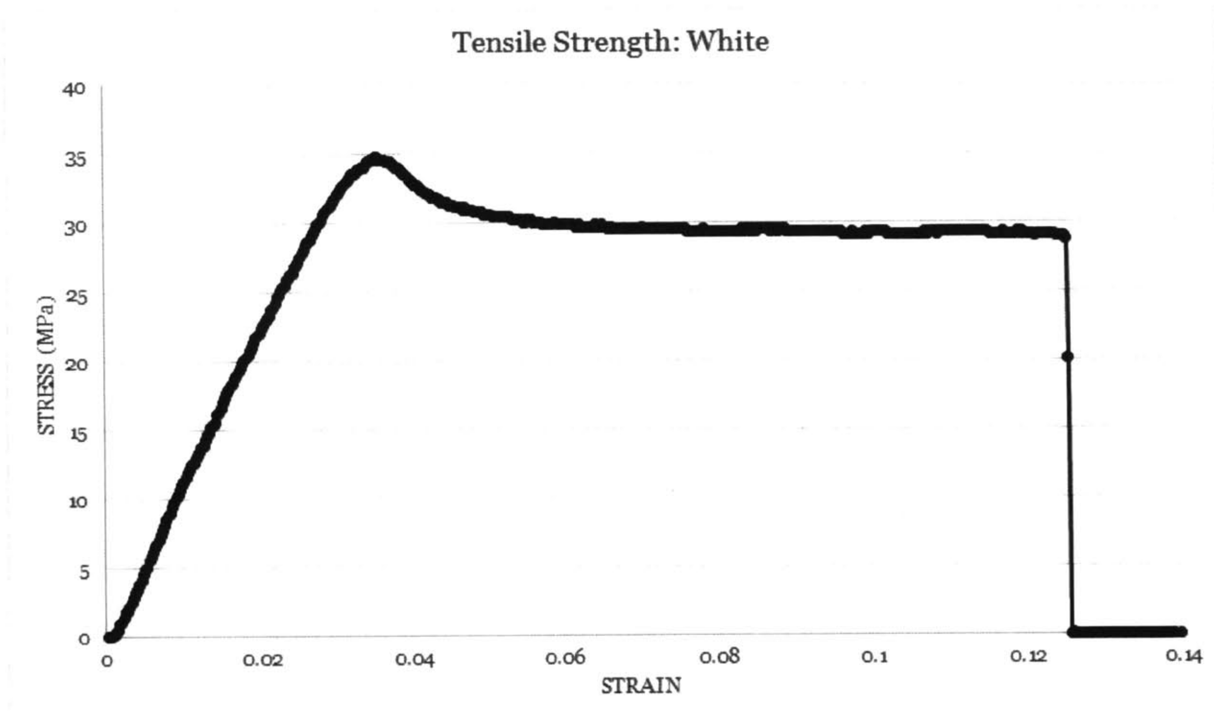

Figure C-E. Tensile Stress-Strain Curve for White PLA. The stress, in MPa, is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain, which is dimensionless, was found by dividing the extension by the body length of the dog bone — 33mm.

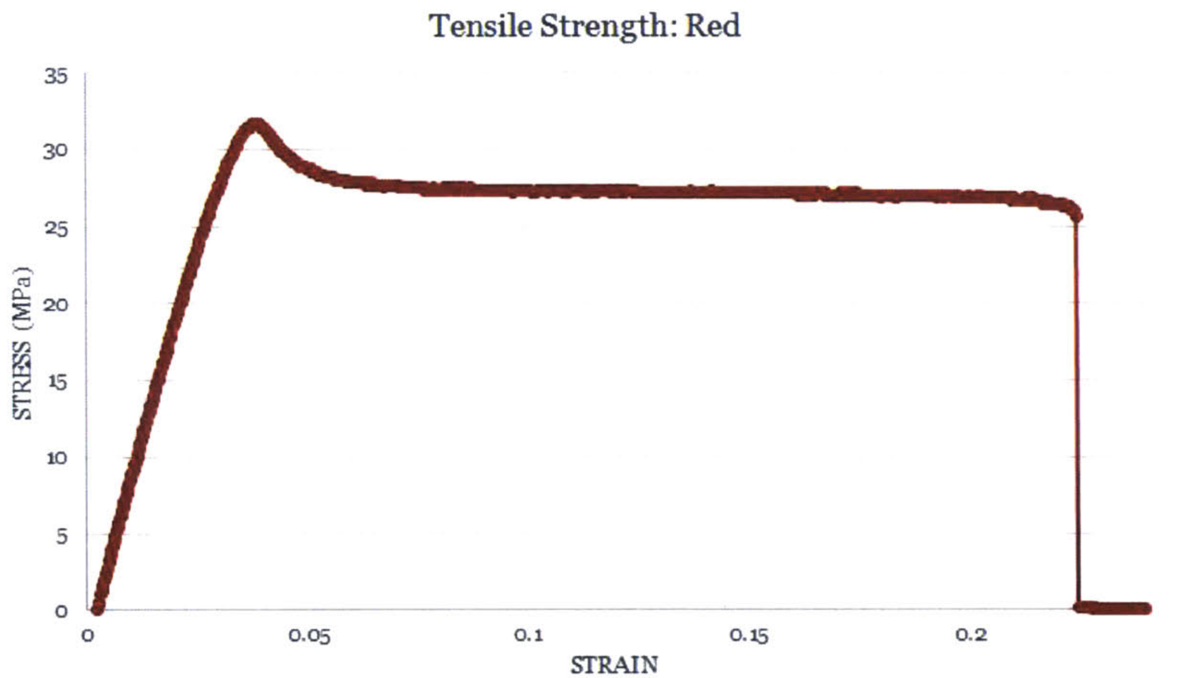

Figure C-F. Tensile Stress-Strain Curve for Red PLA. The stress is found by dividing the load by the cross sectional area of the thinnest part of the dog bone. The strain was found by dividing the extension by the body length of the dog bone — 33mm. Red PLA had the lowest yield stress at 32.7 ± 1 MPa.

Type	Inflated Dim1 (mm)	Inflated Dim2 (mm)	Cross-Sectional Area (m^2)	Gauge Length (mm)	Yield	Fixed Yield
R1	6.88	4.1	0.0000232683	33	757.02	777.15
R2	6.76	4.1	0.0000228327	33	712.73	728.84
Y1	6.6	3.82	0.0000205355	33	837.55	841.58
Y2	6.64	3.83	0.0000207312	33	861.71	865.73
Y3	6.61	3.84	0.0000206918	33	825.47	849.64
B1	6.71	4	0.0000220272	33	757.02	785.21
B2	6.66	4	0.0000218507	33	801.31	785.21
B3	6.68	3.98	0.0000217971	33	797.29	813.39
O1	6.68	3.96	0.0000216729	33	970.44	982.53
O2	6.66	3.95	0.0000215412	33	974.46	966.41
O3	6.68	3.93	0.0000214866	33	994.59998	970.43998
G1	6.66	3.87	0.000021046	33	849.63	853.66

G2	6.58	3.78	0.0000202241	33	845.61	829.51
G3	6.62	3.9	0.0000210945	33	841.58	861.72
W1	6.66	4.06	0.0000222221	33	781.18003	773.13003
W2	6.7	3.99	0.0000219296	33	595.95	672.46
W3	6.64	3.91	0.0000212248	33	797.29	773.13

Table C. Mechanical Testing Table. The dimension lengths, the cross sectional areas, the gauge length, and the yields are listed here. The dimensions in column 2 and 3 are inflated by 0.47 mm which is accounted for in the cross sectional area. The gauge length is from the dogbone being a type IV dogbone by ASTM standards. The yield is corrected by adding whatever the load was after the fracture — which should be zero but isn't because of the weight of the Instron.

6.4 Appendix D:

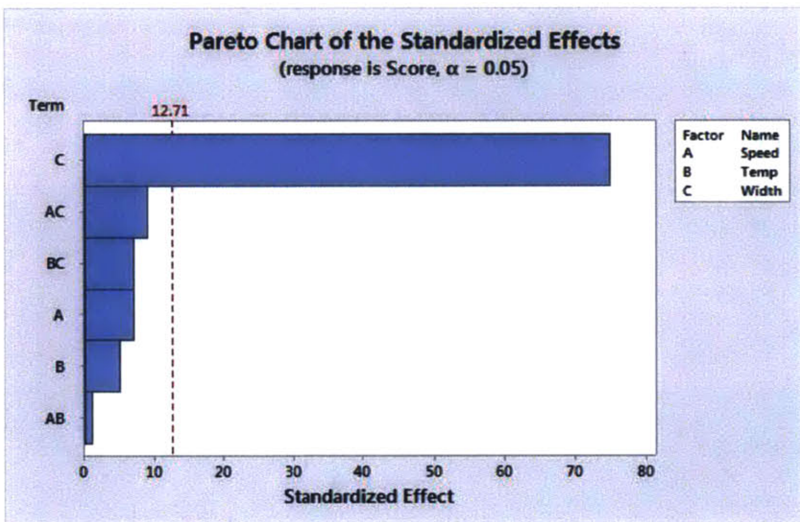

Figure D. Screening Design Pareto Plot for Alpha Calibration Test. This Pareto Plot was done by Paul Burke to figure out which of the three variables were most dominant to save time printing. C, or first layer extrusion width, was found to be the most statistically significant variable.

6.5 Appendix E:

Gray:

ROUND 1

ROUND 1 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.3	190

MEDIUM	0.2	180
LOW	0.1	170

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	6	8	10
2	M	H	7	7	10.5
3	L	H	1	3	2.5
4	H	M	3	9	7.5
5	M	M	8	7	11.5
6	L	M	5	1	1.5
7	H	L	0	2	1
8	M	L	8	5	10.5
9	L	L	11	5	13.5

ROUND 2

ROUND_2 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.18	178
LOW	0.12	173

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	14	2	15
2	H	L	7	9	11.5
3	L	L	4	6	7
4	L	H	0	16	8

White:

ROUND 1

ROUND 1 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.3	190
MED	0.2	180
LOW	0.1	170

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	0	11	5.5
2	M	H			0
3	L	H	3	4	5
4	H	M	6	10	11
5	M	M	3	7	6.5
6	L	M	3	8	7
7	H	L	2	8	6
8	M	L	5	11	10.5
9	L	L	0	0	0

ROUND 2

ROUND 2 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.27	177
LOW	0.23	173

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	1	14	8
2	H	L		9	4.5
3	L	H	1	14	8
4	L	L	1	8	5

Red:

ROUND 1

ROUND 1 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.3	190
MED	0.2	180
LOW	0.1	170

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	4	12	10
2	M	H	6	9	10.5

3	L	H	0	1	0.5
4	H	M	1	8	5
5	M	M	6	9	10.5
6	L	M	0	1	0.5
7	H	L	0	7	3.5
8	M	L	5	9	9.5
9	L	L	0	1	0.5

ROUND 2

ROUND 2 VARIABLES	**Offset**	**First Layer Extrusion Width**
HIGH	0.2	185
LOW	0.15	180

Part ID	**Offset**	**Width**	**Full**	**Partial**	**Total Score**
1	H	H	5	6	8
2	H	L	2	8	6
3	L	L	5	11	10.5
4	L	H	3	10	8

Yellow:

ROUND 1

ROUND 1 VARIABLES	**Offset**	**First Layer Extrusion Width**
HIGH	0.3	190
MED	0.2	180
LOW	0.1	170

Part ID	**Offset**	**Width**	**Full**	**Partial**	**Total Score**
1	H	H	8	8	12
2	M	H	8	7	11.5
3	L	H	0	0	0
4	H	M	8	8	12
5	M	M	7	8	11

6	L	M	0	0	0
7	H	L	5	9	9.5
8	M	L	11	5	13.5
9	L	L	0	0	0

ROUND 2

ROUND 2 VARIABLES	Offset	First Layer Extrusion Width
HIGH	0.22	177
LOW	0.18	172

Part ID	Offset	Width	Printer Number	Full	Partial	Total Score
1	H	H	9	8	8	12
2	H	L	9	13	3	14.5
3	L	L	9	0	4	2
4	L	H	9	0	2	1

Orange:

ROUND 1

Rd 1 Variable	Offset	First Layer Extrusion Width
HIGH	0.3	190
MED	0.2	180
LOW	0.1	170

Part ID	Offset	Width	Full	Partial	Total Score
1	H	H	8	8	12
2	M	H	5	11	10.5
3	L	H	0	4	2
4	H	M	6	10	11
5	M	M	11	5	13.5
6	L	M	0	4	2
7	H	L	6	10	11
8	M	L	8	8	12

9	L	L	0	4	2

Table E-A. Tables for Calibrated Alpha Tests.

Offset and FLEW% Non-linear Relationship with Mechanical and Thermal Tests:

Color	B: FLEW%	C: Offset	D: Score	Product BD	Product BD	Product CD	Product BCD
Orange	180	0.25	13.5	45	2430	3.375	607.5
Yellow	172	0.22	14.5	37.84	2494	3.19	548.68
Grey	174	0.2	14.5	34.8	2523	2.9	504.6
Blue	170	0.25	13.5	42.5	2295	3.375	573.75
White	170	0.2	12.5	34	2125	2.5	425
Red	182	0.3	12.5	54.6	2275	3.75	682.5

Table E-B. Offset and FLEW% Non-linear Relationship with Mechanical and Thermal Tests. It doesn't seem there's a simple proportional relationship between the alpha calibration settings.

6.6 Appendix F:

Delta Test:

ROUND 1:

ROUND 1 VARIABLES	E Multiplier	Acceleration	Speed	Width%
HIGH	1.2	500	140	150
MED	1	300	130	130
LOW	0.8	100	60	110

Table F-A. Settings for Delta Test and their Values.

Part ID	Color	E Multiplier	Acceleration	Speed	Width%
1	Grey	M	L	H	L
2	White	L	M	H	L
3	Orange	M	M	M	H
4	Yellow	M	M	L	M
5	Grey	H	H	M	M
6	Red	H	M	M	L
7	Red	M	H	H	H

8	Yellow	L	H	M	L
9	White	M	L	M	M
10	Orange	h	L	L	l
11	Grey	L	M	L	H
12	Blue	L	L	M	H
13	Blue	M	H	L	L
14	Red	L	L	L	M
15	White	H	H	L	H
16	Orange	L	H	H	M
17	Blue	H	M	H	M
18	Yellow	H	L	H	H

Table F-B. The Settings for Each Print for the Delta Test.

6.7 Appendix G:

Zeta Test:

RD1 VARIABLES	EXTERNAL-PERIMETER EXTRUSION-WIDTH PERCENTAGE	PERIMETER SPEED
HIGH	1.05	130
MED	1	120
LOW	0.95	110

Table G-A. Settings for the Round One Zeta Test for All PLA Colors. The external perimeter extrusion width (EPEW) percentage controls the amount of material extruded for the outermost layer of the 3D printed part. The perimeter speed controls the speed of the extruder as it prints the outermost layer of the 3D printed part.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	L	M	1
2	M	H	4
3	H	M	2
4	L	H	3
5	M	L	—
6	H	L	—

Table G-B. Grey PLA Scores for the Round One Zeta Test.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	L	L	2
2	H	M	4
3	M	H	6
4	M	L	5
5	L	M	5
6	L	H	3

Table G-C. White PLA Scores for the Round One Zeta Test.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	L	L	4
2	H	M	2
3	M	H	5
4	M	L	6
5	L	M	1
6	L	H	3

Table G-D. Blue PLA Scores for the Round One Zeta Test.

PART ID	EPEW	PERIMETER SPEED	SCORE
1	M	H	6
2	H	M	3
3	L	H	2
4	H	L	4
5	L	M	1
6	M	L	5

Table G-E. Yellow PLA Scores for the Round One Zeta Test.

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