An Additive Manufacturing Acrylic for Use in the 32 Tesla All Superconducting Magnet

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AN ADDITIVE MANUFACTURING ACRYLIC FOR USE IN THE 32 TESLA ALL
SUPERCONDUCTING MAGNET

By

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Dedication:

This work is dedicated to my parents who instilled in me a lighthearted approach to excellence

and a desire for knowledge.

And to my wife and my son;

Without whom I would be without a why.
ACKNOWLEDGMENTS

I would like to first acknowledge my wife whose seemingly unlimited patience and perseverance made this possible.

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ABSTRACT

The National High Magnetic Field Laboratory is building a world record all superconducting magnet known as the “32T”. It requires many thousands of parts, but in particular one part called the “heater lead cover” is unusually expensive to manufacture. These parts have been so far made out of a glass filled epoxy known as G-10, and conventionally machined. The proposal in this paper is to change the material and manufacturing method to additive manufacturing using the material called “RGD 430”. If the expensive to machine material is replaced with the automatically printed material, the total cost for the project will be reduced from $21,000 to $455, a 98% cost savings.

To replace the original material, however, the updated material must be able to perform adequately in the designed situation. In order be an acceptable replacement, RGD 430 must survive some minor amount of strain, in a cryogenic environment. It is shown to survive a sufficient amount of strain

The final tolerance for dimensions along the width and length of printed parts is more precise than ± 0.10mm. The final tolerance for the dimensions in the thickness of printed parts is more precise than ±0.25mm. This corresponds to the required maximum tolerance on the heater lead covers of ± 0.10 mm

Before utilizing the material, there should be a few additional tests run on it to ensure it will work in-situ at 4.2K. Those tests are outside the scope of this thesis. It should be noted that the material tested is only one of a great many additive manufacturing materials that are available commercially and at low cost. Any future funded work should take advantage of the readily available products from industry. This thesis serves as a proof of viability for using additively manufactured materials in a cryogenic environment.
CHAPTER ONE
INTRODUCTION

The intention of the document presented here is to show the viability of additively manufactured—commonly referred to as 3-D or “printed”—materials, as engineering materials for 4.2K and high magnetic field applications. To fully characterize a material for all possible conditions in this environment is beyond the scope of a thesis. It is in this light, therefore, that the necessary characterization of the acrylic RGD 430 described in chapter 2 to replace the epoxy and glass composite engineering material garolite G-10/FR4 (known as G-10) in the “heater lead cover” is completed here. This characterization encompasses the material’s molecular composition, its manufacturing method, the macroscopic structure and the options therein, the mechanical properties at room temperature and 77K and the effects of these characteristics on the application of the given part.

Much attention has been given recently to developing high temperature superconducting (hereafter called HTS) magnets. Though the discovery of HTS materials was in the 1980’s, a practical commercial supply has only recently become available [1]. Currently the most promising commercial HTS material is a rare-earth metal based cuprate superconductor known generically as ReBCO. Due to the extreme requirement of ~1-3% angle orientation between grain boundaries in the ReBCO conductor [2] (nearly macroscopic single-crystal texture), these suppliers are only able to deliver flat conductor in the form of tapes. Magnet designers are used to building coils with round wire, or cables. These flat tapes introduce design convolutions, which are not present with round wire designs. Complications in conductor pitch change causes a need for magnets to be wound in the pancake technique [3]. Specifically this technique means that the leads for the protection heaters will not exit axially, as is typical, but radially where there
is minimal area for materials support. This requires the items on the outer radius of the coils to be very thin. This can be seen in figure 1. The thinnest portion of the heater lead is 1.19mm. These heaters leads will rarely if ever be stressed. This is due to the fact that the heaters will only fire if the coil needs to be protected. There are no forces acting upon the heater leads unless the heaters are utilized. However, when the heater leads are used, the conductor will be burdened with some Lorentz forces, which will be sufficiently mechanically supported internally. However there will be some motion. This motion must be absorbed by the heater lead covers.

In the 32T all superconducting magnet, the heater lead covers have been made from G-10 in all the prototypes. This part must be radially thin, and longer than the coil (around 400 mm). Shown in figure 1 is a screenshot of a reduced length version of this part, with a span of only ~120 mm. The cost of this small version using G-10 and standard machining techniques is $420 per part, even when ordered in quantities. The estimated quoted cost for the full-size part which is ~400 mm long is at least $1,500. There will be at fourteen such parts on the finished coil. The cost for mechanical parts is $21,000. Finding a way to reduce cost drove the intent behind this research.

Figure 1: Screenshot of part called "Short Heater Lead Cover"
1.1 Environment

The Magnet Science and Technology (MS&T) division at the NHMFL is currently in the process of developing a high magnetic field all superconducting magnet known as the 32T. This magnet will include many hundreds of engineering drawings, with many thousands of individual parts, all of which are made from a pallet of about a half a dozen materials. These materials include the following:

- The superconductor itself (ReBCO), which is a very complex composite, constituting the bulk of the magnet’s volume, and cost of the magnet; a purchased material
- 316L stainless steel which is used for most of the structural elements
- C10100 copper (or 101 copper), which is known as oxygen free high conductivity copper, or OFHC copper
- G-10, which is a high-performance glass filled epoxy
- 85N, which is a glass-filled polyimide film that is a high performance dielectric
- Trace amounts of the following:
  - 63/37 lead tin solder
  - Alumina powder as a thin dielectric coating on some of the stainless steel
  - Teflon FEP (fluorinated ethylene propylene) as a thin thermoplastic adhesive between polyimide layers

Most of these materials have undergone decades of characterization and proof testing in coils predating even the NHMFL. These materials are the generally accepted ones for use in superconducting magnets. The addition of a new material to the tool belt of the magnet designer
is a rare occurrence. This is often done out of necessity. In this thesis, however, the target is the introduction of a new material to reduce development time and cost.

This additively manufactured acrylic is intended to replace a part made out of G-10 that is very expensive to manufacture.

1.1.1 High Magnetic Field

This material will be subjected to a very high magnetic field on the order of $2 \text{T}$ in a cryogenically-cooled solenoid. These high magnetic fields can alter electronic band properties, the electronic orbitals, the conduction properties, the dielectric strengths, and various other materials characteristics. For polymers however, there is very little effect. There is some evidence for the magnetic torque on polymers from the magnetic field increasing crystallinity during processing [4], but at cryogenic temperatures there is not enough diffusion for this torque to have any effect.

The field will not affect the heater lead covers directly. It will induce some force in the heater lead conductor that is in contact with the heater lead covers. This force causes a strain in the conductor, which will be translated through to the heater lead covers. The heater lead covers will therefore only be indirectly affected by the magnetic field.

1.1.2 Cryogenics

Cryogenic conditions that will be incurred by this sample material are liquid nitrogen (77K), liquid helium (4K), and occasional moderate vacuum at these temperatures. There should be minimal effects due to the liquid itself, as helium is a noble gas and nitrogen is quite inert. Reduction in temperature for polymers typically changes many of their mechanical characteristics. A summary of several polymers is given by Reed and Walsh in the paper
Tensile Properties of Resins at Low Temperature [5]. Figure 2 shows the elastic modulus and tensile strength for cyanoesters and polyester, which is the most similar of these to the RRGD 430 material tested here. The data are shown for the temperatures from 295K, to 77K and 4K.

Figure 2: Tensile strength vs. Young's modulus for cyanate esters and polyester [5]

G is a Down Chemical developed cyanate ester
I is a Rhone-Poulenc developed cyanate ester
P is an Owens Corning developed polyester
Q is an ICL developed cyanate ester
The exact definitions for G, I, P and Q are given in Appendix A
This graph shows a strong grouping of modulus to temperature, and sometimes of strength to temperature. From these data and the work in ref. [5] my working assumption was the material would experience brittle failure at 77K and 4K. Tests at 77K confirmed this assumption. Tests have not been done at 4K due to budget restrictions but RGD 430 is also expected to be brittle at 4K.

1.2 Additive Manufacturing

Additive manufacturing is generally known as rapid prototyping, or 3-D printing. In the past almost all final stage manufacturing was subtractive. Steel foundries supplied billets or plates or round stock, that was parted off and otherwise manipulated by subtracting material until a final retail or commercial product was created. Additive manufacturing changed this.

Additive manufacturing began in the 1980s. The first techniques included fused deposition molding, and most importantly photopolymer stereolithography. The process of Stereo lithography classically is the method of taking a bath of polymer, and then applying an ultraviolet light to it in a computer aided pattern based off of a CAD file. The UV light will excite the electrons in the polymers, and allow them to activate and grow together, or polymerize. The bath of liquid is then filled up slightly higher so that a following layer can be built upon the first layer. This process is repeated until a finished part is removed from the polymer bath.

There is another method called plaster printing (PP) which is a powdered plaster that is applied as a very thin layer, and then inkjet style print heads come along in a raster pattern, and apply a binder. The raster pattern is one where areas are sprayed by very closely spaced rows, and each pass moves the printer head up one column. After a layer is complete, another flat layer, encompassing the entirety of the 3-D printer bed is applied.
There are many other methods, including some that utilize coppers, steels and even refractory metals like niobium [6]. One can use lasers, electron beams, furnace or selective heat sintering, to bring materials above their glass transition temperature, or close to their melting point, so that they can be easily molded or shaped from grains, or wires, or extrusions into the final desired shape.

The additive manufacturing process described in this thesis is a hybrid between stereolithography, and the raster style printer method. This method is described in more detail in chapter two.
CHAPTER TWO
METHODOLOGY

2.1 Sample Design

The design of the samples was developed with assistance from Robert Walsh at the National High Magnetic Field Laboratory. The original cryogenic polymer tensile specimen design was developed at the National Institute for Standards and Technology. It is shown in figure 3 from a paper entitled “Tensile Properties of Resins at Low Temperatures” [4].

Figure 3: Original double reduced area sample design for polymer measurements. Dimensions are in mm. Thickness dimensions are not given.
This design utilizes two area reductions for the purpose of ensuring a standard specimen cross sectional area and test section length. This design was modified for use in a small cryostat. In the original design in figure 3 proved to be too long for the small cryostat. So in order to reduce the length of the specimen the portions of the design that are flat have been eliminated.

2.2 Sample Processing

2.2.1 Objet 30 – The Additive Manufacturing Machine

The Objet 30 is an additive manufacturing machine (a 3-D printer), located in and belonging to the High Performance Materials Institute (HPMI) at Florida State University. It was originally purchased from Objet, but that company has since been bought out by Stratasys Systems. Stratasys Systems currently offers an Objet 30 Pro, which according to telephone discussions with the company, is merely a software upgrade to the original Objet 30. The machine can utilize eight different photopolymer materials.

The Objet 30 utilizes an altered stereolithography method. Instead of using the traditional polymer bath, and then UV curing the polymers in layers, the machine uses inkjet like printer

Figure 4: Custom design of the 25.4 μm thick short double-reduced tensile sample
heads to apply a thin layer 16μm thick, and then immediately applies ultraviolet light to cure the polymer in place. The objet has the advantage over many 3-D printers, in that it utilizes a secondary material. This “support” material is used to allow for parts to be produced with overhangs, and holes that require support, so they do not collapse during printing. The heater lead covers in figure 1 have several details that require overhangs to be of good quality.

The Objet 30 can build objects up to a maximum dimension of 294mm x 192mm x 148.6mm. It has a listed accuracy of ± 0.1mm, which is tested and described in detail in chapter three [6]. The accuracy of the dimensions through the thickness depends on following the appropriate procedures for manufacturing.

The first step of printing any samples is to clean the printing heads themselves. These print heads as discussed in chapters three and four, are critical to creating repeatable parts. The print heads are cleaned with a lint free cloth, and a spray bottle of isopropyl alcohol, until there is no more discoloration upon wiping. The print head also has a hot roller for leveling surfaces; it must be cleaned in the same manner.

The second step of printing is the purging of the material fill lines, heads, heaters and all other flow sections. The Objet 30 does this automatically by selecting “Reinitiate Wizard”. This also warms the heads up for use. The heads and material must be around 78°C to print properly. This takes approximately 15 minutes.

While the previous step is being automatically completed, the next step can simultaneously take place. The organization of the items to be printed must occur. It accepts “.stl” files, which are a readily available form of CAD file that all popular CAD software programs can output. The software program can manipulate these drawings in a number of ways, including scaling, rotating, translating, creating specific surface finishes etc. The main purpose is
to serve as an intermediary between a single design, and the platform or “Tray” that these parts are printed on. After laying out the tray for printing, one simply hits “Build Tray”, and another automatic wizard controls the process from there.

The Objet 30 is a multi-material additive manufacturing machine. It is capable of printing both a strong polymer, and a very weak wax-like polymer to act as a support material. This support material makes the Objet 30 capable of making parts with holes, or overhangs, or any other complicated geometry that the NHMFL might encounter.

2.2.2 RGD 430 – The Material

RGD 430 is a thermoplastic acrylic that is UV cured that is used in the Objet series of printers from Stratasys Systems Inc. It was chosen because it has the lowest Young’s modulus of the materials available for the Objet 30, and thus the assumption is that it will be able to absorb more strain at cryogenic temperatures.

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM</th>
<th>Units</th>
<th>Metric</th>
<th>Units</th>
<th>Imperial</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>D-638-03</td>
<td>MPa</td>
<td>20-30</td>
<td>psi</td>
<td>2800-4350</td>
</tr>
<tr>
<td>Elongation at break</td>
<td>D-638-05</td>
<td>%</td>
<td>40-50</td>
<td>%</td>
<td>40-50</td>
</tr>
<tr>
<td>Modulus of elasticity</td>
<td>D-638-04</td>
<td>MPa</td>
<td>1000-1200</td>
<td>psi</td>
<td>145,000-175,000</td>
</tr>
<tr>
<td>Flexural Strength</td>
<td>D-790-03</td>
<td>MPa</td>
<td>30-40</td>
<td>psi</td>
<td>4350-5800</td>
</tr>
<tr>
<td>Flexural Modulus</td>
<td>D-790-04</td>
<td>MPa</td>
<td>1200-1600</td>
<td>psi</td>
<td>175,000-230,000</td>
</tr>
<tr>
<td>HDT, °C @ 0.45MPa</td>
<td>D-648-06</td>
<td>°C</td>
<td>32-34</td>
<td>°F</td>
<td>90-93</td>
</tr>
<tr>
<td>HDT, °C @ 0.82MPa</td>
<td>D-648-07</td>
<td>°C</td>
<td>32-34</td>
<td>°F</td>
<td>90-93</td>
</tr>
<tr>
<td>Izod Notched Impact</td>
<td>D-256-06</td>
<td>J/m</td>
<td>40-50</td>
<td>ft lb/inch</td>
<td>0.749-0.937</td>
</tr>
<tr>
<td>Water Absorption</td>
<td>D-372-08</td>
<td>%</td>
<td>1.5-1.9</td>
<td>%</td>
<td>1.5-1.9</td>
</tr>
<tr>
<td>Tg, °C</td>
<td>DMA, E’</td>
<td>°C</td>
<td>35-37</td>
<td>°F</td>
<td>95-99</td>
</tr>
<tr>
<td>Shore Hardness (D)</td>
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<td>Scale D</td>
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<td>Scale D</td>
<td>74.78</td>
</tr>
<tr>
<td>Rockwell Hardness</td>
<td>Scale M</td>
<td>Scale M</td>
<td>no data</td>
<td>Scale M</td>
<td>no data</td>
</tr>
<tr>
<td>Polymerized density</td>
<td>ASTM D792</td>
<td>g/cm³</td>
<td>1.15-1.17</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ash content</td>
<td>USP281</td>
<td>%</td>
<td>0.10-0.12</td>
<td>%</td>
<td>0.1-0.12</td>
</tr>
</tbody>
</table>

Figure 5: Stratasys Systems Inc. datasheet for room temperature properties of RGD 430 [6]
Stratasys did not convey the more precise details on the manufacturing of this polymer. Thus things like the viscosity of the preset fluid, the void characteristics, kinetics and suppression, the final molecular weight were not available. In fact, only the information that isobornyl acrylate forms less than 40 percent of the weight of the monomer precursor is given anywhere in the literature. Shown below is an excerpt from the MSDS showing that the majority of the material is proprietary:

<table>
<thead>
<tr>
<th>CAS #</th>
<th>Component</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>5888-33-5</td>
<td>Isobornyl acrylate</td>
<td>15-30; 30-40</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Acrylate oligomer</td>
<td>10-30</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Acrylic monomer</td>
<td>10-30</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Acrylic monomer</td>
<td>5-10; 10-30</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Acrylic monomer</td>
<td>5-10; 10-30</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Acrylic monomer</td>
<td>0.1-1.0; 1-5</td>
</tr>
<tr>
<td>Proprietary</td>
<td>Photoinitiator</td>
<td>0.1-1.0; 1-5</td>
</tr>
</tbody>
</table>

Figure 6: Stratasys Systems Inc. component list from the MSDS datasheet for RGD 430 [7]

Isobornyl acrylate is shown below as a schematic:

Figure 7: Isobornyl acrylate [8]
It contains the isomer isoborneol, in the acrylic monomer, replacing the vinyl hydrogen. Giving something akin to the following schematic:

Figure 8 shows a monomer of isobornyl acrylate. The monomer is a vinyl. This means it is effectively an ethylene molecule with one of the hydrogens replaced with some other functional group. This functional group is borneol and is shown in figure 8 as the large connected network of spheres. The black represent carbon, the red represents oxygen and the white are the hydrogens. To polymerize this the double bond between the carbons. It can be seen that there will be good hydrogen bonding between the finished polymers, as the borneol has a more rigid three dimensional structure than the classic thermoplastic polyethylene, which has only a 1 dimensional structure.
2.2.3 Sample Cleaning and Preparation

After the samples are printed as described above, the samples must be removed and prepared for testing. The samples are initially scraped off of the surface of the tray with a putty knife and then set aside. The tray itself is cleaned with further scraping, and a liberal application of isopropyl alcohol and standard lint-free Kim wipes.

After the samples were taken from HPMI to NHMFL, they were mechanically scraped with a soft plastic scraper to remove the support material. As described in chapter four, some of the samples were cleaned with isopropyl alcohol, which turned out to be a very bad way to clean the samples. It reduced the strength of the material by almost 50%. The appropriate method is to simply mechanically remove the excess support material with a soft piece of plastic. Shown in figure 9 is an example of a 0.25mm thick sample, with the support material (a), partially removed and (b) completely removed. This cleaning does cost some man hours, but the expense has not be included in the cost comparisons discussed. The author believes the time is negligible, and on the order of 5 minutes per part.

Figure 9: Printed sample during cleaning. This shows the thickness of the material and the support material. a) shows the partially removed material, and b) shows all of the support material removed.
After cleaning, the samples were measured with calipers, flat anvil micrometers, and ball micrometers as described in chapter three. An example of a cleaned and prepped sample clamping area is given below in figure 10. There are clear striations and random thickness defects shown. These occurred during the printing process.

![Figure 10: Detail of a cleaned tensile sample](image)

Figure 11 shows examples of both striations along the width (a and c) and along the length (b).

![Figure 11: Detail of three prepared tensile samples with striations going along width and length](image)
2.3 Sample Testing

The tensile samples were tested using an MTS (Mechanical Test Systems Corporation) Criterion 42, a liquid nitrogen test cryostat, a custom made extensometer; bolt style grips and an alignment tool. The alignment is critical to minimize the torque encountered by the samples.

The coefficient of thermal expansion was measured using an MTS dilatometer. The system was originally created for testing sample expansion in a controlled furnace, but has been retrofitted to do cryogenic applications. The samples are cooled to 4K in liquid helium, and then allowed to gradually warm up to room temperature.

2.3.1 Test Equipment

The alignment tool—shown in figure 12 below—was used to make sure that the stress passes through the cross section of the material, and not at some unknown angle, which would put an unknown torque on the sample. The samples were placed by aligning the sample edges with lines on the baseplate.

Figure 12: Alignment tool with bolt style clamps and tensile sample in place
The samples were then placed in the MTS Criterion 42. A custom-made test cryostat was created. All available cryostats were too small to allow for individual’s hands to reach in and apply the extensometer. So the cryostat was made from an available polystyrene container.

The extensometer used was a custom-built aluminum-framed sensor. It is the lightest weight extensometer available in the NHMFL materials characterization laboratory for cryogenic use. The choice of this extensometer was so that the additional weight and torque of the extensometer would not cause errors in the readings. This specific extensometer was called the “Shepic” named for the watch craftsman who built it for the NHMFL. It has no official calibrations, so calibrations must be performed in-situ. It is a basic clip-on style device, with a sensor arm connected to a four wire linear output strain gauge. The strain gauge uses a full Wheatstone bridge configuration.

The dilatometer is a Unitherm Model 1101 Dilatometer system with a retrofitted cryostat. The cryostat and all cryogenic portions were designed by the MS&T Materials Testing and Characterization group.

2.3.2 Equipment Calibration

The MTS Criterion 42 has internal verifications for its load cell, which were utilized at the start of every test day. The custom made extensometer required some in-situ calibration. The extensometer was placed on a very large handle-style dial caliper, with G-10 extensions passing down into the liquid nitrogen, where the extensometer was set with the initial length of exactly one inch. This one inch length is the minimum head distance of the extensometer. The dial caliper was then rotated until it reached the maximum expected extension of 0.254 mm (which would give 10% strain). The reading on the MTS Criterion 42 software package were then iteratively changed until they read exactly 0.254 mm extension when rotated, and then 0.00 when
rotated back to zero. The proportional $V_{in}$ to $V_{out}$ setting was recorded and set as -0.0022 (unit less V/V).

The dilatometer uses the timed difference between the heights of two Invar bars. The invar bars are in contact with the sample, and a known copper sample as a standard, to account for any thermal gradients that might occur. The measured ends of the Invar bars are at room temperature. Invar is notable for having an extremely low coefficient of thermal expansion, so its length change due to the temperature difference from room temperature to the sample temperature is negligible. The samples are at first bathed in liquid helium until they reach thermal equilibrium at 4.2K. Then the temperature is allowed to naturally increase through heat transfer through the cryostat walls, floor and invar bars themselves. This creates a moderately slow temperature increase, which is recorded along with the height difference between the invar bars. The nominal temperature ramp rate is less than 1K/minute. However, because it is an uncontrolled system, the maximum can approach 3K/minute. The calibration is done continuously using the copper standard to give an appropriate calibration.

2.3.3 Test Procedure

A total of 48 individual samples were made to be tested for this project. The first ten were tested at room temperature. They had results similar to the stated results, but there was error due to a lack of a procedure. At the end of the room temperature testing a procedure was developed. It was perfected by sample 35.

Samples were printed and prepared as explained in section 2.2. These samples were then measured using the methods described in section 3.1. These samples were then individually placed on the alignment tool shown in figure 12, and bolted into the bolt style clamps. The edges of the sample were aligned visually using parallel lines on the surface of the alignment tool.
The sample was placed into the cryostat and connected with pins through the clamp. The load cell measurement on the MTS machine was set to only read the load due to the weight of the lower clamp (approximately 7 N). The clamps were set in the approximate center of the pin length. When the extensometer was used, this was the time it was placed. The extensometer measurement arm was compressed into the minimal position so that $L_0$ would be exactly 1.00”. Some of the samples were tested without compression of the extensometer. The MTS machine’s top arm was lowered to eliminate the possibility of increasing the load due to thermal contraction when cryogens were added.

Liquid nitrogen was then added according to NHMFL safety procedures. After the clamps stopped boiling nitrogen, a time of 60 seconds was waited to ensure thermal equilibrium in the sample. Then the test was initiated and computer controlled.

The MTS machine moved the clamps at a speed of 0.5 mm/minute. This was a single cycle strain to failure, where the sample is pulled on until it completely fails. Data for the extensometer, load cell, and total extension were continuously recorded, and saved for later analysis. The samples fractured. The cryostat was drained into an awaiting dewar. The clamps were heated with a heat gun, until the ice melted. The samples were taken out and cataloged for later analysis. The procedure was started over with placement in the bolt style clamps and a new sample.

2.4 Data Analysis

Data analysis was done mainly using Microsoft Excel. There were additional calculations done by hand. The statistical analysis histograms were created using a Microsoft add-in known as “Data Analysis: Histogram”.

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The magnetic field solver used is an internal macro-based Excel document known as “SolenO” developed at the NHMFL. It solves the difficult hyperbolic functions for magnetic field to an accuracy of at least 4 significant digits.

Error bars throughout the document indicate either standard deviation or sample maxima/minima of measurements. The measurement error was considered too small be relevant, as measurement error is much smaller than random variation. Standard deviations were on the order of 0.5-15%, whereas measurement errors of the calipers and micrometers were on the order of 0.1%-0.5%.
CHAPTER THREE
GEOMETRIC TOLERANCING

In the engineering division of the Magnet Science and Technology department, engineering drawings conform to a strict set of standards. There are secondary and occasionally tertiary checks on these drawings to make sure they are correct before leaving the lab for manufacture. One of the most critical parts of these is the geometric tolerance. These are the physical size and dimension requirements that a machinist or manufacturer must adhere to. The three standard tolerances on a linear length, width or thickness vary from the loose ± 1 mm, to the typical ± 0.25 mm, to the precise ± 0.10 mm. Any tolerance that needs to be more precise than ± 0.10 mm requires a special callout per dimension. Anything more relaxed than ± 1 mm is put in parenthesis () to emphasize that the dimension is more of a recommendation. There are other more rare callouts such as straightness, thread fit, concentricity, and others. These have not been examined in detail in this research due to their rarity. The straightness in the sample area of the tensile specimens was measured, and found to have a straightness tolerance maximum of 0.010 mm over a 25.4 mm length ( 0.00040” over a 1” length), which is a very good tolerance.

All the samples printed for testing were measured for geometric tolerances. In general these samples conformed to the desired dimension. The desired dimensions are described here as a “nominal engineering” dimensions, and this should be defined as the dimension that was drafted into the CAD software. Parts with these nominal engineering dimensions were produced in the profile of the printer, which is the printer’s xy plane. This is layer width and length of the sample. However the thickness of each piece seemed to be slightly variable. This variation was a skewed thickness distribution where the thickness was generally averaged just above the nominal engineering thickness but then skewed thicker. This could be intentionally setup by the
manufacturer for the purpose of ensuring that all thicknesses are at least the nominal engineering thickness if not greater, so that if there were strength requirements they would be met.

3.1 Method of Measurement

All measurements were taken by hand. There were 42 tensile samples and 6 thermal contraction samples. The widths of the tensile samples were measured in five places with the calipers described below in table 1. The width of the caliper heads are approximately 3.5mm. Since the sample section is 25.4mm long, the five measurements did not entirely encapsulate every position along the length of the sample. It was noticed that there were visible thickness variations along the samples either along the length of the specimen, or across the width of the specimen. This was the reason for measuring both the peaks of the thickness with flat anvil micrometers and the details of the thickness with ball micrometers. These micrometers are described below.

Thickness measurements with the flat anvil were taken at the same five locations as the width measurements. As the width of the flat anvil is 6.35mm, this should have taken into account all areas of the profile with some overlap.

Thickness measurements with the ball micrometer (ball mic) were taken at the same five locations as with the width and flat anvil micrometer. There were additional measurements at the bottom, middle and top of the sample, along with width. The ball micrometer has some radius approximately 6.35 mm. As this micrometer approximates the measurement of the thickness from point to point, it should—and does—give some smaller measurement than the flat anvil. Also, the ball micrometers are more likely to cause elastic deformation in the samples due to the small surface contact area, which will lead to some further reduction in the measured thickness.
This effect has been ignored due to the unknown force exerted by the ratcheting mechanism of the ball mic, and the indeterminate surface contact area between the ball and the sample.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Model Number</th>
<th>Instrument</th>
<th>Name</th>
<th>Measurement Range</th>
<th>Minimum discretization</th>
<th>Factory Calibration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mitutoyo</td>
<td>500-196-30</td>
<td>Standard Digital Caliper</td>
<td>Caliper</td>
<td>0-6 inches</td>
<td>.0005 inches (0.01 mm)</td>
<td>Yes</td>
</tr>
<tr>
<td>Mitutoyo</td>
<td>293-831-30</td>
<td>Ratchet Stop Flat Anvil Micrometers</td>
<td>Flat Anvil</td>
<td>0-1 inch</td>
<td>0.00005 inches (0.001 mm)</td>
<td>No</td>
</tr>
<tr>
<td>Mitutoyo</td>
<td>293-831-30</td>
<td>Ratchet Stop Ball Micrometer</td>
<td>Ball mic</td>
<td>0-1 inch</td>
<td>0.00005 inches (0.001 mm)</td>
<td>No</td>
</tr>
</tbody>
</table>

3.2 Geometric Statistics

3.2.1 Profile Statistics

It was found that the profile dimensions were generally within the desired precision tolerance of ± 0.1 mm. Figure 13 shows the 5.08mm thick tensile sample’s width distribution. The vertical line on the left is the desired nominal engineering width of 5.08 mm. The line on the right is the average of all the width measurements. This sufficiently approximates a unimodal Gaussian distribution, and as such it has a standard deviation of 0.030 mm. The average deviates from the nominal a total of + 0.031. Thus for a 99.7% confidence in the width, a good model would be:
This would give a minimum of 5.02mm and a maximum of 5.2mm for a nominal engineering width of 5.08mm. This 5.2mm is out of tolerance for a precision tolerance as described above, but if this positive offset of the average is taken into account, it will be within tolerance. Thus if an engineer desired a 5.08mm wide piece on average, the should be programed as 5.05, so that the resultant part will measure 5.08 ± 0.090mm with a 99.7% confidence that all dimensions will be within tolerance.

However, if the desired outcome is stress related, then adhering to the nominal engineering dimension will be desired. It would seem that the Object manufacturers knew of this statistical variance, and programmed the machine to give a positive offset, so that all minimums would approximate the desired dimension. Therefore it should be an engineering practice that dimensions that are stress originated should be designed exactly, and those that have a strong geometric requirement where stress is not critical, should be designed with the above modeled offset.

Equation 1: 99.7% confident model for the dimensional tolerance

\[ D_{Dimension Measured} = (D_{Dimension Nominal} + O_{Average - Nominal}) \pm 3 \times \sigma_{Standard Deviation} \]

\[ D = D_o + O \pm 3 \times \sigma \] (1)

Equation 2: Empirical model for dimensions in the x-y plane

\[ W_{Width Measured} = (W_{Nominal} + 0.03mm) \pm 0.09mm \] (2)
The samples made for the tests of the coefficient of thermal expansion are rectangles with dimensions 5x5x50 mm. They followed the same pattern as the tensile sample width measurements above. There are fewer measurements so the sample standard deviation is higher. This measurement seems also to be a bit skewed positive, but again there are few sample numbers (30 total measurements). This has a standard deviation of 0.035 mm. The average deviates from the nominal to + 0.025mm. Thus for a 99.7% confidence in the width a good model would be:

Figure 13: 5.08mm thick tensile sample width distribution
\[ W_{\text{Width\,Measured}} = (W_{\text{Nominal}} + 0.025\,mm) \pm 0.105\,mm_{(3)} \]

Equation 3: Less confident model of dimensions in the x-y plane due to fewer sample measurements

Figure 14: 5mm width measurement distribution

This would give a minimum for a nominal engineering width of 5mm of 4.92mm, and a maximum of 5.13mm. This 5.13mm is out of tolerance for a precision tolerance as described before, but again, it can be well modeled and should be taken into account for any critical design. Samples measurements with unusually large thicknesses were checked to ensure that all of the support material had been removed, so that these are the actual material thicknesses.
The coefficient of thermal expansion samples also had length measurements taken. There were only a total of 6 of these measurements possible, but they still conform to the previous two in-plane models.

![50mm Length Distribution](image)

Figure 15: 50mm length distribution

The average is exactly 0.030 mm above the nominal. The standard deviation is 0.0179 mm. This difference from the previous models in equations two and three is most definitely due to there being so few samples taken.
3.2.2 Thickness Statistics

The statistical variation of the geometric tolerancing through the thickness of these samples was also modeled, but had much stronger variance than the profile statistics shown in the above section. This dimension correlates to the $z$ plane in the printer. To begin with, the simpler example of the thickness of the coefficient of thermal expansion samples is plotted below in figure 16. The average has increased and the deviation is obviously wider. However this does not fit a standard Gaussian curve, as it looks bi-modal. One could argue that there are only 30 samples, and thus a Gaussian fit is sufficient as long as statistical confidence is not discussed. Thus a back of the envelope model for this system would be

$$T_{Thicknes Measured} = (T_{Nominal} + 0.07mm) \pm 0.168mm_{(4)}$$

Equation 4: Empirical Model of Dimensions in the $z$ Plane

This is derived from the standard deviation being 0.056mm and the difference between the nominal and mean is $+0.070mm$. However, this scatter is not due to random variation. This scatter is position dependent in some places along the profile plane of the printer. This is shown below in figure 17 where in the measurements of the CTE sample thicknesses where there was a clear pattern of samples 4, 5 and 6 being thicker than the average thickness. This was preceded by a smooth transition where sample 3 had an intermediate thickness between the lower and upper numbered samples. This smooth transition bodes well for any engineering strengths associated with these larger thickness variations. This also shows up later in the tensile sample measurements. It is very repeatable, and could be modeled if desired.
Figure 16: 5mm thickness distribution

Figure 17: Surface plot of sample thickness and position
The tensile samples show similar characteristics with the thickness variations described above. The statistical analysis will not be by sample position, as there are 42 samples with 20 measurements each, which are 840 individual measurements. In general these statistics will be discussed using the model described above where the offset is the difference from the nominal engineering thickness to the mean of the sample set, and the standard deviation is around the mean. The data is also shown to be skewed positive, and this will be discussed. The worst case of thickness variations is in figure 18. This specimen was used for room temperature measurement. In the sample the thickness variation striations are shown across the width as shown in figure 18a. The yellow lines are approximate continuations of these striations. Detail 18b shows a defect only seen in three samples. The deformation is outside the sample area but it is worth noting. These deformations were in identical pieces, printed right next to each other.

Figure 18: Detail of a) striations across width, and of b) a rare anomaly
These samples also exhibited some variation in the thickness, which is not only measureable, but visible. Figure 19 shows the striations along the length of the sample and figure 19a shows the noise, or random thickness variation.

Figure 19: Detail of a) random variation in the thickness, and b) striations along the length of the sample
This variation, most visible to the left of detail a in figure 19, does not dominate the thickness variations, as will be shown in upcoming sections.

As was described before when the model was built for predicting the actual thickness for a nominal engineering thickness, there is some offset of the average from the nominal.

Figure 20: Tensile sample thickness offset of mean from nominal
Shows no correlation to thickness itself
This offset average for all of the sample thicknesses from the nominal is shown in figure 21. These are given in terms of the nominal engineering thickness, which shows that there is no variance dependence on the thickness itself. However there is a clear dependence on the thickness with sample number as shown below in figure 21. Clearly the samples from 10-23 have large variation compared to the rest of the samples. The only correlation that is known from the lab notes taken during manufacture was that these samples were made first, and that the printer heads were not cleaned as described in chapter two. The assumption is that the heads were inappropriately cleaned, and thus lead to large variations in the thickness of the material. It can clearly be seen that the thickness normalizes after sample #23, which is when the author began cleaning the heads himself. After that variation dropped significantly. This issue propagates throughout the rest of the testing and analysis. The stress in particular was affected by this wild variation, as is shown in chapter four. Thus it is recommended that thorough cleaning of the heads be done before every printing session.

It can also be seen that the striations travel in straight lines along in the y axis of the printer surface. This is visible in every sample made, as small striations that were recorded as either along, or across the width of the sample. These striations were thickness variations, and were recorded with the ball micrometer to give sufficient detail for analysis. These were assumed to cause some stress dependence.

The thickness variations have been analyzed below for sample number 39. Figure 23 shows the thickness variation of sample 39, which was the worst for all samples. The bottom of the sample was arbitrarily chosen as one of the edges along the one inch length of specimen section. The middle is the center of the specimen section, and the top is the opposite edge from the bottom edge. The variation was continuously increasing from the bottom to the top, but it was always
worst on the top portion of the sample. This is shown in figure 22. This is the positioning of the tensile sample measurements throughout the thesis.

Figure 21: Distribution or spread of offset of mean from nominal

Figure 22: Positioning of tensile sample measurements showing top, middle, bottom measurements along the width, and position numbers along the length of the specimen area. Shown at the right are the striation directions.
Sample number 39 had the worst width variation. It varied from a minimum of 1.25mm to a maximum of 1.43 mm, giving a 14% variation. Figure 24 shows a three dimensional surface graph of that thickness. The axis labeled position across length corresponds to the y axis of the printer, or the length of the specimen. The axis labeled position across width corresponds to the x axis of the printer or the width of the specimen. The thickness is the z axis of the printer. These variations should be eliminated. After discussions with the manufacturer, appropriate cleaning should eliminate this error.

Figure 23: Average thickness across width for the worst thickness variation sample
In the more than 600 individual measurements made, maximum deviation from the nominal measurement was thicker than the nominal engineering thickness by 0.245 mm (0.0096”). This is still within the limits of the standard or “typical” tolerance. Figure 25 shows the distribution of individual thickness measurements deviation from the nominal engineering thickness. The ball micrometer measurements are centered on the histogram bin of -0.01 to 0 mm offset, and are fairly strongly skewed positive. However out of all the 600 measurements taken 41 were over the precision tolerance mentioned above and zero were below the precision tolerance. Therefore it can be stated that there is a greater than 93.2% probability that a printed
thickness will be within the precision tolerance and that probability is \( P > 93.2\% \). Thus multiple confidence empirical models could be given to the designer for the thickness measurements.

\[
T = T_o \pm 0.10\text{mm} \quad (5)
\]

Equation 5: 93.2\% confident empirical model for tolerance of thickness

\[
T = T_o \pm \begin{bmatrix} +0.25\text{mm} \\ \text{or} \\ -0.10\text{mm} \end{bmatrix} \quad (6)
\]

Equation 6: > 99\% confident empirical model for tolerance of thickness

![Figure 25: Distribution of deviation from nominal engineering thickness](image)

Figure 25: Distribution of deviation from nominal engineering thickness
A designer must understand and apply mechanical stresses and materials properties to keep the design from failing. The main mechanical material constraints in machine design are the strength and elastic modulus of the material chosen. Anything where the stresses applied are negligible is described as a kinematic situation. A situation in which the stresses involved are not negligible is described as a “machine design”. The design of the heater lead covers described in chapter five requires that for the entirety of their lifecycle these parts will undergo a maximum strain only once, which is due to the heaters being fired. After that the coil is disassembled and rebuilt. Thus mechanical fatigue cycling is not a concern. As mentioned in chapter two, a single-cycle strain to-failure-method was used. Brittle failure was seen at cryogenic temperatures. Ultimate tensile strength was calculated using a number of methods. These methods were numerous due to the spread in cross sectional area as described in chapter three.

4.1 Young’s Modulus

Young’s modulus is the measure of stiffness of a material. For a given linear stress, the modulus is the change in length of some portion with a constant cross sectional area.

\[ \sigma = E \varepsilon \]  (7)

Equation 7: Generic equation relating stress, strain, and Young’s modulus
Where $\sigma$ is the stress due to a load normal to some cross sectional area divided by that area, $\varepsilon$ is the change in some sample length divided by the original length, and $E$ is the Young’s modulus. The higher the value of $E$ the stiffer the material is. See table 2 for engineering moduli for some common materials used at cryogenic temperatures.

Table 2: Linear elastic moduli for commonly used cryogenic materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Modulus at room temperature ~ 300 K (GPa)</th>
<th>Modulus at 77K (Liquid nitrogen) (GPa)</th>
<th>Modulus at 4K (Liquid helium) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Steel 316L</td>
<td>193</td>
<td>193</td>
<td>193</td>
</tr>
<tr>
<td>Copper</td>
<td>17.3</td>
<td>20</td>
<td>22</td>
</tr>
<tr>
<td>Garolite G-10/FR4</td>
<td>18-20</td>
<td>19-32 [10]</td>
<td></td>
</tr>
<tr>
<td>RGD 430</td>
<td>3-5</td>
<td>9-12</td>
<td></td>
</tr>
</tbody>
</table>

Judging from Table 2 the modulus of the RGD 430 seems to be similar to for the Copper and G-10, as long as the materials are in a cryogenic environment. From the ASM Engineered Materials Handbook on Engineering Plastics [4], it is known that the modulus of polymer based materials varies with temperature. As the temperature drops below the glass transition temperature, the modulus increases many fold. See figure 26 for details:
This chart suggests that for polymers at cryogenic temperatures the modulus should approach \( \log(E \text{ (Pa)}) \approx 9.7 \) and thus \( E \approx 10 \text{ GPa} \). However, for Garolite G-10 this fails to be true. The value of the modulus is above 10 GPa for G-10 due to the ionic bond strength of the S-2 glass in the composite. As shown below, this relation for \( E \approx 10 \text{ GPa} \) seems to be valid for RGD430 at 77K.
The decision to choose RGD 430 was based on figure 26 above. The RGD 430 had the lowest glass transition temperature and the lowest modulus for all available materials that could be used on the Objet 30. This meant that it would possibly remain ductile until a lower temperature. If the modulus stays low the stress caused by the controlled strain in the heater leads will remain low, which will ideally not cause fracture until a higher strain is incurred. Figure 27 shows the spread of the data for Young’s modulus at 77K for all samples of RGD430 where strain was measured. E is given as a function of nominal engineering thickness to show that there is no apparent correlation to thickness.

![Figure 27: Young’s modulus for RGD 430 at 77K](image-url)
Shown in figure 28 is the distribution of moduli at 77K. This shows a spread of the measured moduli of between 9-12 GPa. For design purposes the average is given as 10.2 GPa. It is the opinion of the author that there is some error in these moduli. Due to the strain being measured by an extensometer, the initial length is intended to be exactly one inch. The extensometer was calibrated as described in chapter 2, and it is believed that this calibration is correct. However, when placing the extensometer in place it can be applied with an initial length different than that which is intended. If put in the exact position of $L_o = 1.00”$ then it is mechanically set at it’s zero point. This is the calibrated zero point. In this way it cannot be reduced to $L < 1.00”$. However, due to the initial design—see chapter 2 for details—the extensometer must require very little force to move some extension, otherwise it would affect the measurement of the material it was trying to characterize. Therefore it takes very little effort to extend the $L_o$ to greater than 1.00”. So in this way some of these measurements have an $L_o > 1.00”$. This means then that some of the moduli have a lesser value than what is true. Thus the author states that the modulus should be reported as 10-11 GPa at 77K.

This modulus is half of what G-10 is the same temperature. However this does not disqualify RGD430 as an engineering material. If applied to the application of heater lead cover as described in chapter five, then it may simply need a larger cross sectional area. Since the heater lead cover will experience strain dependent motion (meaning that the motion of the conductor underneath will not be changed much by the presence of the heater lead cover itself), then the smaller modulus is an advantage, as that will reduce the stress accrued within the cover.
4.2 Stress

The failure strength of a material is the most critical mechanical aspect of any machine design. A confident characterization should encompass: isotropy, processing characteristics, processing history, ambient conditions and any additional conditions that might change the strength characteristics of a material.

Figure 28: Distribution of modulus from all samples available
4.2.1 Engineering Stress

The mechanical strength test data is best broken down into the three phases of testing as can be seen in figure 29. These phases correspond to the following:

- **Phase 1:** Initial phase where the samples were tested with the striations across the width (see figure 22 for reference).
- **Phase 2:** Samples were tested with striations along the length, but had very low strength values. This is due to cleaning with isopropyl alcohol.
- **Phase 3:** Samples were reprinted from phase 2, and cleaned only mechanically to give more accurate results.

The simplest stress calculation is that of the nominal engineering stress, which is the load at divided by the nominal engineering thickness and width. The engineering stress at failure is the load at failure divided by the initial area. For stating a conservative value the nominal area is given as the product of the minimum of both the thickness and width as measured Thus:

\[
\sigma_{\text{Engineering@Failure}} = \frac{P_{\text{Load@Failure}}}{A_{\text{nom}}} 
\]

Equation 8: Engineering stress at failure
A function of load at failure and the nominal engineering area

In phase one the samples were printed with the intention of testing in two phases. The first with the striations across the width, and then print some samples with the striations along
the length of the sample specimen. Phase one turned out quite nicely, it had an average engineering stress at failure of 141 MPa, and a standard deviation as a percentage of the average stress at failure of 6.54%. However, in the middle of testing phase two, it became obvious that something had gone quite awry. Sample 23, which was the first test of phase two, came out at 45 MPa, which was almost as weak as the room temperature samples. After spending some time going over the notes, it was found that the samples were cleaned with isopropyl alcohol prior to being tested. This was the only difference except the striation direction. There was the use of a solvent caused degradation that led to a reduction in the average strength of 40%. This was the purpose for testing yet again for phase three; to accurately measure the striations across the length.

Figure 29: Nominal engineering stress at failure for RGD 430 at 77K
Phase three was a retest of phase two, but with only mechanical cleaning of the samples. This showed a minor reduction in average strength, down to 122 MPa, but as will be shown in the coming sections, this is to be expected. Only data from phase one and phase three are used for stress characteristics. This gives an average stress at failure of 116 MPa, and a standard deviation of 18.7 MPa.

4.2.2 Material Stress

The more accurate method for measuring the material stress is to modify the calculation method above to give a different stress output. The cross sectional area as described in detail throughout chapter three varies. The variations in the width of the sample are negligible; however the thickness of the sample can vary from the nominal by a maximum measured difference of 0.245 mm (0.0096”). Thus to get an accurate characterization of the material strength an accurate model for failure must be developed.

For those samples where the striations occurred across the width of the sample space, the cross sectional area is not constant. It varies along the length of the sample space. This variation will lead to failure in a very specific position, where the minimum thickness is observed. Thus the material stress at failure for phase one samples should be described as:

\[
\sigma_{Material@Failure} = \frac{P_{Load@Failure}}{A_{minimum}} \quad (9)
\]

Equation 9: Material stress at failure for RGD430 at 77K printed across the width
In the other case of the thickness variations occurring along the length of the sample space as shown in figure 22, the more accurate description would be of an average thickness. This is true due to the fact that the cross sectional area is generally constant throughout the length of the sample. As shown in see figure 17 describing sample 39 in chapter three. Thus for an accurate model of the material stress where the striations occur along the length of the sample, the equation should read:

\[
\sigma_{Material\@Failure} = \frac{P_{Load\@Failure}}{A_{Average}}
\] (10)

Equation 10: Material stress at failure for RGD 430 at 77K printed across the width

This model is not as accurate as the stress in equation 9, since the load applied will not be symmetric, and will induce some unknown torque on the material, which will give a higher local strain and thus higher load and thus much higher stress through the thinner portion of the material. This gives a stress distribution shown in figure 30 in light grey.

This reduction in failure stress shown in Figure 30 for the striations perpendicular to the length of the sample, leads to the conclusion that the samples with the striations along the width give the more conservative data for calculation of the strength of the material in cryogenic
applications. Thus with 99.7% confidence it is concluded that the material strength is greater than 113.5 MPa at 77K.

![Calculated Material Stress at Failure Distribution](image)

Figure 30: Distribution of calculated material stress at failure
The most accurate data is in dark blue, with the striations across the width
Minimum Material Strength

4.3 Strain at Failure

One of the often used measurements in cryogenic machine design is percentage strain to failure. This can also be used in a kinematic situation where the material in question does not experience any of the appreciable strain. Thus the designer needs to ensure that the material can survive the strain. For example, the superconductor ReBCO coated conductor to be utilized in
the 32T project has a well-known irreversible strain (or strain at failure) of around 0.6%. At 77K
the strain at failure of the RGD430 was measured and is described by the distribution in figure
31. This spread gives a 99.7% confidence that the strain at failure will be greater than 0.92%.
Compared to the superconductor it is in contact with, the strain at failure is sufficiently large for
most coil applications made with ReBCO coated conductor.

Figure 31: Distribution of strain at failure for RGD 430 at 77K

4.4 Fracture

The fracture mechanism is very relevant to the design consideration. The fracture mode
for each sample was cataloged and recorded, unless the fractured specimen broke into many
pieces that fell into the liquid nitrogen bath. There were two kinds of fracture, typical tensile
brittle fracture, and brittle fracture due to torque. The typical brittle fracture was always seen in the samples with the striations perpendicular to the length. This is illustrated below in figure 32.

The fracture begins at one edge and propagates through the reduced area, then takes a 45 degree angle—which is typical of brittle fractures—and continues down another valley between striations that has a reduced area.

The fracture due to torque was always seen in samples with the parallel to the length of the sample. These fractures are characterized by a common break along one edge, and then as above the fracture continues down a valley between striations. This time it travels down a certain direction down some length, and then grows in the other direction. This spreads the failure area out and causes the pattern seen below in figure 33. There is a missing area where the failure area spread out.
4.5 Shock Loading

The thermal shock resistance of RGD 430 was tested. In the literature many polymers cannot survive the thermal expansion and contraction within themselves, and shatter due to temperature gradients. This was tested by submerging samples into liquid nitrogen, and then air warming. The samples survived multiple cycles. The samples were then submerged in liquid nitrogen for several minutes, and then immediately immersed in water. They survived multiple cycles. Then the samples underwent cryogenic impact studies, where they were submerged in liquid nitrogen for several minutes and then propelled forcefully against a marble table top immediately after exiting the cryogenic bath. The samples survived several cycles, with no failure. Finally the samples were allowed to stay in the nitrogen bath for several minutes, upon which they were quickly evacuated to an insulating surface of G-10 and robustly impacted with increasing force by a hammer. They survived two impacts. The third shattered the sample. The author believes that this is sufficient testing to show that thermal gradients, and impact loading are not issues with this material for this particular application as a heater lead cover. It would be a good idea to do fracture toughness tests in the future to determine a value for $K_{ic}$.
4.6 Coefficient of Thermal Expansion

One of the critical aspects of cryogenic design is the fact that materials will decrease in spatial dimensions as the temperature decreases. This reduction in size per Kelvin change in temperature is described as the coefficient of thermal expansion (CTE). The common units for the CTE are $K^{-1}$, however for most cryogenic materials this reduction in size is reported for a change in length of a sample in terms of percentages. This percentage difference is for the total change from 295K to 4K. This percentage for copper is on the order of 0.3% [7]. For the ReBCO superconductor in the coil, it is measured as 0.315% reduction from 295K to 4K [8]. For the G-10 that the RGD430 is intended to replace, the CTE from 295 to 4K is anisotropic. G-10 has woven glass fibers in different directions, and they cause changes in the thermal expansion and contraction. The percentage reduction from 295 to 4K is 0.25% in the warp direction, and 0.7% in the normal direction [14]. As shown in figure 34 this thermal expansion is well characterized for RGD430 and nearly 4 times larger than for copper or the superconductor:

This shows that there is a 1.8% increase in linear size from ambient to operating conditions for RGD430. This is significantly larger than the copper or the conductor, but since it is known about, it can be taken into account in the design. Depending on the geometry of the situation it can be mathematically modeled, or compensated for in the “scale” of the component.
Figure 34: Thermal expansion of copper and RGD 430 from 4.2K to 295K
CHAPTER FIVE
APPLICATION TO THE HEATER LEAD COVER

The heater lead cover itself will undergo a strain controlled situation. That is the strength of the heater lead cover should not affect how far the conductor under it will travel mechanically.

Figure 35: Prototype Coil known as 20/70.
Figure shows detail of a) superconducting coil with all hardware, b) cross sectional area of coil with heater lead on the right hand side, c) the heater lead itself, and d) the heater lead profile with axial positions (in mm) from the midplane of the coil

The values that determine the travel of the conductor is the radial magnetic field magnitude and direction, the heater current magnitude and direction, and the effective modulus
of the conductor. The radial magnetic field is shown along the length of the conductor below in figure 36:

![Figure 36: Worst case of radial magnetic field at the position of the heater lead covers](image)

This model was developed with the use of the program called Soleno. The worst case scenario is when the maximum current is going through the heater lead with the longest length. Which from the coil design makes the scenario 20 A in the heater lead, with a 312 mm long piece of conductor. The force exerted on the conductor is given by:

$$\vec{F}_{Lorentz} = \vec{I} \times \vec{l} \times \vec{B} \quad (11)$$

Equation 11: Lorentz force
Where $B$ is the radial field in figure 36, $I$ is the maximum current of 20 A. These are field and current values are vectors that are perfectly perpendicular, thus the cross product will be orthogonal to both of them, and equal in magnitude to the scalar product of the two. This is shown in terms of force per length as follows in figure 35:

![Figure 37: Load distribution applied to the conductor](image)

To find the strain and see if the heater lead with RGD430 will survive, the deflection must be calculated. The deflection is a function of the forces and moments through the body of the conductor. The sum of forces and moments with both ends soldered in position to steel or copper leads gives maximum resultant distributed load on the conductor of 0.00462 N/mm. If this were a worst case scenario then: the effective force was for some reason would be 0.00462 N/mm throughout the half length of the conductor, from the top flange to the mid plane, then this would give a total load of 0.7N. Therefore the maximum deflection encountered from equation 12 would be around 0.1577mm:
Where \( \delta \) is the deflection, \( q \) is the constant load (0.00462 N/mm), \( L \) is the length of the applied load (half the total length \( 312\text{mm}/2 = 166\text{mm} \)), \( E \) is the modulus of the conductor \((E = 165 \text{ GPa}[9])\) and \( I \) is the moment of inertia through the thickness \((1.755 \text{ mm}^4)\).

This deflection could easily be absorbed by the 3-D Printed RGD 430 at cryogenic temperatures, as long as the width of the pieces was over 17mm, or 0.67". The current design calls for pieces to be made that are 12.7mm wide; this adjustment could be made quickly and easily.

Equation 12: Calculation of deflection of superconducting heater leads in a worst case scenario
Equation for a beam simply supported on both ends with constant load [15]
CHAPTER SIX
CONCLUSIONS

6.1 Review

The following characteristics were found for RGD430 at 77K:

- Brittle Failure
- Yield strengths $\geq 100$MPa
- Elastic Modulus $\geq 10$GPa
- Strain at failure $\geq 0.92\%$
- Due to printing process:
  - Repeatable thickness variations
  - Geometric tolerances of $\pm 0.10$mm in the xy plane and +0.25/-0.10mm in the z direction of the printer

6.2 Discussion

The majority of the characterization needed to apply this material as the heater lead covers in 32T has been completed here. There is some additional work that could be done in the future. That is discussed in the following section.

RGD 430 fell within the expected bounds of thermoplastics as described in the literature for cryogenic applications [11]. Its modulus and strength increase with decreasing temperature.

The only concerns seen throughout this testing sequence was the strength reduction due to the use of isopropyl. To mitigate this wax support material can be removed manually, and no solvent is needed. As discussed in the following section, this should be looked into further.
6.3 Open Characterizations

There are several areas that should be studied before RGD430 is used in any cryogenic system for extended lifecycles. The characterization at 4K must be done. It was not done in this study due to budget constraints. The modulus is expected to increase, but the strength is potentially going to decrease going from 77K to 4K [5]. See figure 2 to show that the failure strength is reduced from 77K to 4K for many polymers. This may be detrimental to the strain at failure, which is critical to apply RGD430 for use in the heater lead covers.

This material is somewhat porous, and may absorb water during thermal cycling to room temperature. The expansion of water upon freezing could compromise the strength of the material. It may not have been the isopropyl as described in chapter four that caused the damage, but the water itself. The material listed can absorb up to 2% water in a short period of time. That water may freeze and cause damage to the RGD430 material. This should be examined.

As described above, the fracture toughness would also be a critical test to run, as this material always has brittle failure at cryogenic temperatures.

There will be as much as a 200 V differential between the conductor inside the heater leads and the material outside of the heater leads. Therefore dielectric breakdown in vacuum—also known as Paschen testing—should be performed.

There are other characterizations that would be very helpful if performed. The outgassing of this material, as it is somewhat porous, would be a good thing to know, as the 32T will undergo some vacuum pumping before helium cool downs.

If one were interested in utilizing this material inside of the coil winding pack itself, then many other tests should be done, including resistivity, thin layer dielectric breakdown, thermal conductivity as a function of temperature and specific heat as a function of temperature.
There are dozens of other 3-D printed materials that are available that should be considered. In this thesis a single material has been chosen, purchased and tested. It was shown to be a viable candidate for low strain, or rapid prototyping applications in cryogenic environments. For the Objet 30 alone, there are 7 other similar materials that could be used. There are additively manufactured steels, and ceramics, copper alloys, aluminum alloys and many more materials available from commercial vendors with equally outstanding tolerances and lead times.

It would in fact, not be outside of the scope of the MS&T department to develop 3-D printed materials specifically for the cryogenic high field environment. A few ideas for potential research candidates would be:

- Non-magnetic austenite stabilized stainless steels
- Highly cold worked and highly twinned copper for pulsed and resistive magnets
- Printed polyimides as super-insulators, with extremely high dielectric breakdown voltages
- Klegecel foam substitutes as baffles for lowering thermal heat loss to cryogenic baths
**APPENDIX A**

**RESIN IDENTIFICATION IN CRYOGENIC TESTS**

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<tr>
<th>Type &amp; Designation</th>
<th>Description</th>
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<td>Dow Chemical</td>
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<td>Shell</td>
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<td>102</td>
</tr>
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*The resins are identified to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials identified are necessarily the best available for the purpose.*
REFERENCES


BIOGRAPHICAL SKETCH

Zachary Johnson is a Research Engineer in the 32T project in the department of Magnet Science and Technology at the National High Magnetic Field Laboratory. He has Bachelor’s degrees in both Business Management and Mechanical Engineering. His graduate research is done in addition to his engineering work at the NHMFL. All of this is done in addition to his being a father and a husband.