ABSTRACT

TAMBEY, PRASHANT. Comparison of Mechanical Properties of Additively Manufactured Polyether Ether Ketone (PEEK) and Compression Molded PEEK Utilized as a Bone Plate. (Under the direction of Dr. Mark Pankow, Dr. Ola Harrysson and Dr. Denis Marcellin - Little).

The objective of this study in the field of Additive manufacturing is to study the feasibility of 3D printing medical implants with Polyether ether ketone (PEEK) as the material using a cost effective Fused Filament Fabrication process. PEEK implants are currently manufactured by Selective Laser Sintering (SLS) method which is very expensive.

The medical implant chosen to be investigated in this study is a cortical bone plate and the 3D printer used is the first ever commercial FFF PEEK printer, HPP 155 manufactured by Apium Additive Technologies GmbH. The control samples were machined out of PEEK sheets manufactured by compression molding. The testing method and creation of samples were done in accordance with ASTM F382-14, Standard Specification and Test Method for Metallic Bone Plates. The bone plates were mounted on simulated bones made of Delrin® and attached using 3.5 mm cortical screws. Bone plate samples were tested in bending strength and bending fatigue and compared to control to determine if the mechanical properties are comparable and is the additively manufactured PEEK is a good substitute for making bone plates and other medical Implants.

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Comparison of Mechanical Properties of Additively Manufactured Polyether Ether Ketone (PEEK) and Compression Molded PEEK Utilized as a Bone Plate

by
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DEDICATION

This thesis is dedicated to Aai, Baba and my sister for being my pillars in life.
And my elementary and high school teachers for instilling that fiery passion towards science and research which led me to where I am in life today.

BIOGRAPHY

The author was born in Madhya Pradesh, India. He spent his whole childhood in Hyderabad (Andhra Pradesh) where he completed his schooling at Bharatiya Vidya Bhavan's Public school. He then studied at the Indian Institute of Technology - Banaras Hindu University in Varanasi and graduated with his bachelor's degree in Mechanical engineering in 2012. He worked for three years in Maruti Suzuki India limited where he was in the Ride, Handling and Brakes testing department. He quit his job in June 2015 to pursue his Masters degree at North Carolina State University in Mechanical Engineering.

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I would like to express gratitude toward each of my advisors, Dr. Mark Pankow, Dr. Ola Harrysson and Dr. Denis Marcellin-Little. Their invaluable input and support was a major reason for my success in this sparsely researched topic of additive manufacturing of bone plates using PEEK. I would like to thank Dr. Pankow for his constant support and knowledge of testing methods and analysis. I would like to thank Dr. Harrysson for guiding me in the field of additive manufacturing and medical devices which was very new to me. I would like to thank Dr. Marcellin-Little for his vast knowledge on possible practical research areas involving PEEK and how to integrate them with medical devices. Thanks to all the lab members at the BLAST lab and CAMAL lab who always took the time out to listen to my research and provide help.

I would like to thank my family for their support and faith even from the other side of the globe and my oldest friends Rohit, Preeti and Ruby for always being there irrespective of geographic location. I owe my sanity the new friends I made after coming to a totally new country and to the wonderful lifelong friends I met through the Adventure club.

I owe special thanks to Dr. Harvey West who taught me a lot about testing of medical devices, the ASTM standards associated with them and how I could best analyze and present my results.

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1 INTRODUCTION

1.1 Background

In recent years Polyether ether ketone (PEEK) is an important thermoplastic biomaterial that is slowly gaining acceptance in the field of orthopedics and for other medical applications [1]. It belongs to the polyaryl ether ketone (PAEK) family and is known for its high chemical inertness and resistance to temperature. Traditional manufacturing methods for PEEK include injection molding, compression molding and extrusion [2]. The raw material for these operations usually is powdered PEEK or PEEK in the form of pellets. While these methods of production are suitable for industrial purposes which operates on high volume and low customizability it poses many drawbacks in the biomedical field which depends on customizable implants and complex designs.

Additive manufacturing (AM) addresses these shortcomings and is the perfect solution for creating customized implants. The only problem with AM is that it offers only two processes, namely Selective Laser Sintering (SLS) and Fused Filament Fabrication (FFF) to produce PEEK parts. This study uses an FFF based 3D printer for PEEK manufactured by Apium industries GmbH. This study builds on the conclusion of many research papers that 3D printing of PEEK could promote new medical application for PEEK based materials [9].

1.2 Literature Review

1.2.1 PEEK in medical applications

The use of PEEK for medical applications began in the late 1980s and the applications have mostly been on the orthopedic side. The earliest research on PEEK implants was larger in amount on spinal implants. There is ongoing research on it being used in orthopedic bearing and hip stem material [3]. PEEK has been combined with materials like carbon fiber, Barium sulfate (To create image contrast PEEK for imaging purposes) [4] and also coated with biomaterials like hydroxyapatite (HA) or a HA and titanium mixture [15] to improve osseointegration.

Extruded PEEK infused with carbon fiber has been used to make Bone screws and pins which offer similar mechanical properties to titanium or implantable Co-Cr alloys [4]. These also do not interfere with imaging and help get a better knowledge of how the bone is healing.

Spinal Implants

PEEK was introduced in spinal cages in 1990s and was used for stabilization of the anterior column of the lumbar or cervical spine and facilitates their fusion [3]. One of the major advantages of having a PEEK cage over metallic cages is the ease in visualization of the recovery of the area using common radiographic techniques. The reduced implant stiffness also helps as it is more similar to the stiffness of the bone around it which reduces stress concentrations and promotes healing [4].

Cranial Implants

Using PEEK for cranial reconstruction surgery is a recent development. The reasons for choosing a PEEK implant are same as the ones for spinal implants like greater biointegration, lower infection rates and easier implant techniques [5]. The implant is unique and made using SLS additive manufacturing technique and is manufactured by only one company Oxford Performance Materials (OPM). It got its FDA approval in 2013 and it is sold under the product name OsteoFab®.

Bone plates and Hip/Knee Implants

One of the concerns of having a metallic fracture fixation device is the reduction in bone quality in the area adjacent to the bone plate, due to stress shielding [3, 6]. Because the bone plate becomes the primary load bearing structure the bone even after healing weakens. Because the elastic modulus of PEEK is similar to the cortical bone it can help overcome this problem [10]. The disadvantage of a thermosetting polymer is that contouring it to the shape of the bone fragments is difficult but with additive manufacturing this can be addressed. There are various studies regarding the use of Carbon reinforced PEEK and other types of PEEK as bone plate material but there are no clinical reports of the performance of these bone plates [3, 7].

Orthopedic stems completely made of PEEK are still being researched but composite stems which are coated with PEEK have already gotten FDA approval as early as 2006 [3]. It is called the Versys Epoch Fullcoat stem. It has a forged CoCr alloy inner core and has an intermediate coat of PEEK and an outer bone in-growth later made of pure Ti fiber metal.

In one study PEEK and Carbon Fiber Reinforced (CFR) PEEK have been compared to Ultra High Molecular Weight Polythene (UHMWPE) which is the standard bearing surface for a total knee replacement [8]. This study concluded that PEEK and CFR-PEEK had much higher wear rates compared to UHMWPE and there was also evidence of cracking and material failure observed in the contact region.

1.2.2 PEEK processing methods

Injection molding

This process is very suitable for mass production of PEEK implants and components. It uses PEEK pellets or powder which is fed into a hopper. They melt in a heating zone and are pressurized. This molten PEEK is then ejected into a set of heated molds which form the implants. This process is only good for high volume production because of the high cost of creating molds. The temperature in the heating area needs to be in the range of 400°C and the mold needs to be around 175 - 205°C [2]. Mold temperature and the rate of cooling of molded parts plays an important role in determining the amount of crystallinity in the implant which determines its mechanical properties.

Compression molding

Compression molding is a process used to create sheets or plates of a material. The heated press contains two heated plates. The lower plate is fed with PEEK pellets or powder which

melts due to the heat. The two plates are then pressed together and cooled while maintaining the pressure. It is typically used for low volume processes like prototyping or components with thick cross sections.

Extrusion

This is a manufacturing process used to produce long stock shapes such as bars, rods and sheets. The process is similar to injection molding but the only difference here is that the molten PEEK is forced through a heated die instead of a mold and as it cools it moves ahead along the line of extrusion.

Machining

Machining is a post processing method to create required parts from products made by the above mentioned processes. Machining processes on PEEK are prone to propagating residual stresses so annealing is recommended to relieve all internal stresses before machining it. A second annealing can be done in case of heavy machining operations which might have caused localized heating [2].

1.2.3 Additive manufacturing of PEEK

Additive manufacturing (AM) is a general expression for several computer-based methods to transform CAD data into physical objects. Joining materials based on a 'layer wise' approach is the common denominator for different AM-techniques [12]. Due to extensive use in the medical industry and stability in mechanical properties up to about 240°C, additive manufacturing of PEEK is a very lucrative prospective but because of its high melting point of around 343°C [2] 3D printing it becomes a challenge. There are only two processes available to 3D print PEEK material.

Selective Laser Sintering

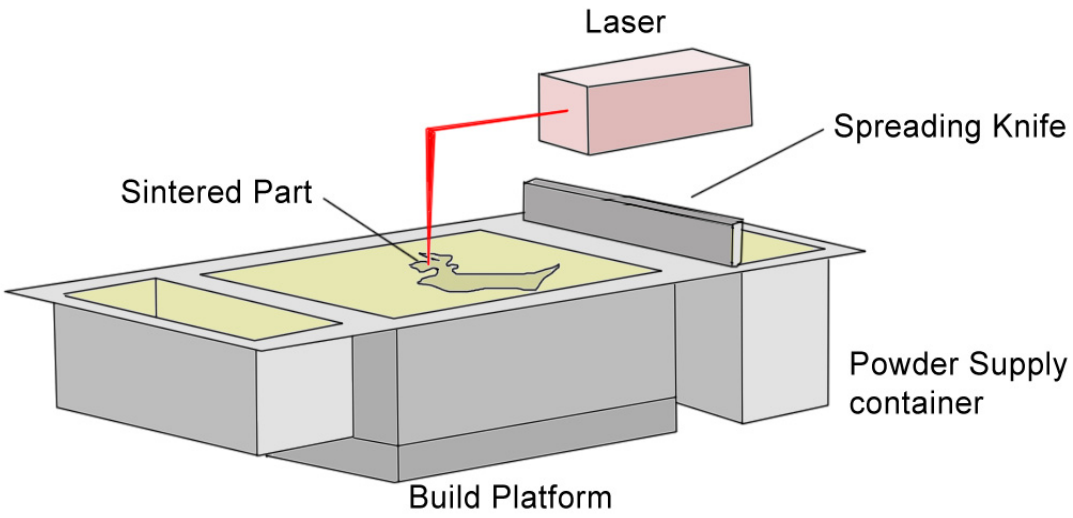

Figure 1-1: Principle of SLS

This layer based additive manufacturing process makes use of powder as its raw material. The process of manufacturing starts with inputting the CAD data of the part to be made into

the controller. This data is sliced up into layers and is sent to the machine. The laser selectively sinters the powder in the topmost layer according to the sliced layer data. After sintering of one layer is finished the platform is lowered and an even coat of powder of uniform thickness is applied over the sintered layer [11]. The whole setup is preheated and is done in a chemically inert environment.

The only commercially available SLS printer for PEEK is the EOSINT P800 and its cost is upwards of $ 400,000. The manufacturer also makes their own grade of PEEK powder called PEEK HP3 but there are companies that have successfully used other PEEK powders to create medical grade implants. Oxford Performance Materials (OPM) is one such company that manufactures medical grade cranial, facial and spinal implants using a different PEEK powder.

The major drawback of SLS manufacturing method is the cost involved.

Fused Filament Fabrication

This layer based AM process makes use of a spool of filament as its raw material. This filament is passed through a heated nozzle which sits on two movable rails giving it 2 degrees of motion. As the print nozzle finishes one layer the print bed moves down as per the set layer height and the printing of the second layer starts.

Most commercially available FFF based 3D printer nozzles only go up to 300°C. PEEK melts at 343°C and also the temperature of the build chamber and print bed needs to be kept higher than 100°C. These are the reason why very few manufacturers have tried to print PEEK [12].

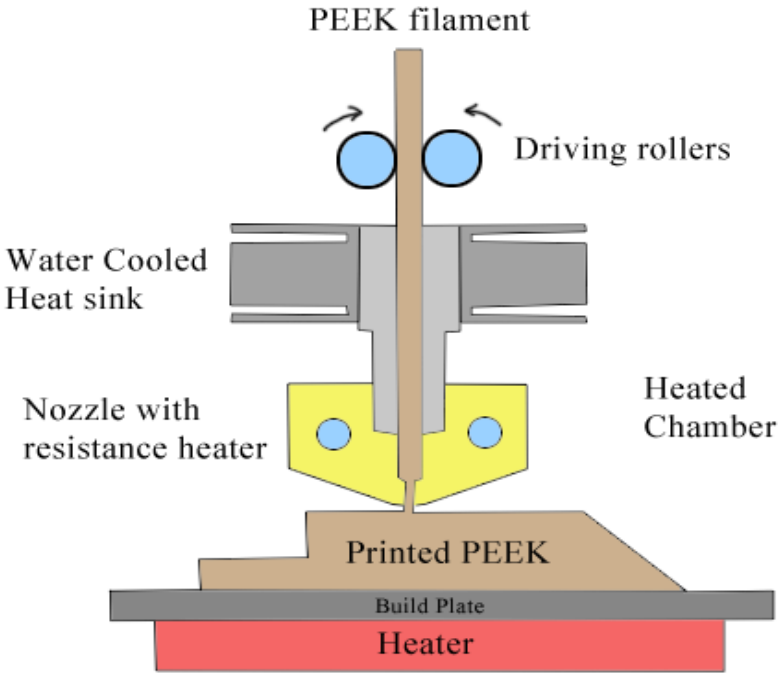

Figure 1-2: Probable setup for filament based PEEK printing

One of the first commercially available FFF based PEEK printer is made by a Germany based startup Indmatec GmbH which has been since renamed as Apium Industries GmbH. The heated nozzle in their machine HPP155 can be heated up to 420°C and the bed can be heated to 120°C. It has an attached zone heater around the heated nozzle which maintains the temperature of the build area. The material used in the filament is Victrex™ PEEK.

They are in the process of making a new printer which can directly print implant grade PEEK. The mechanical properties of parts manufactured from this PEEK printer have been researched in this study. At a price of $ 20,000 the HPP 155 is a great way to make cost effective PEEK parts.

Another new China based company called INTAMSYS has recently unveiled its line of PEEK printer in April 2017.

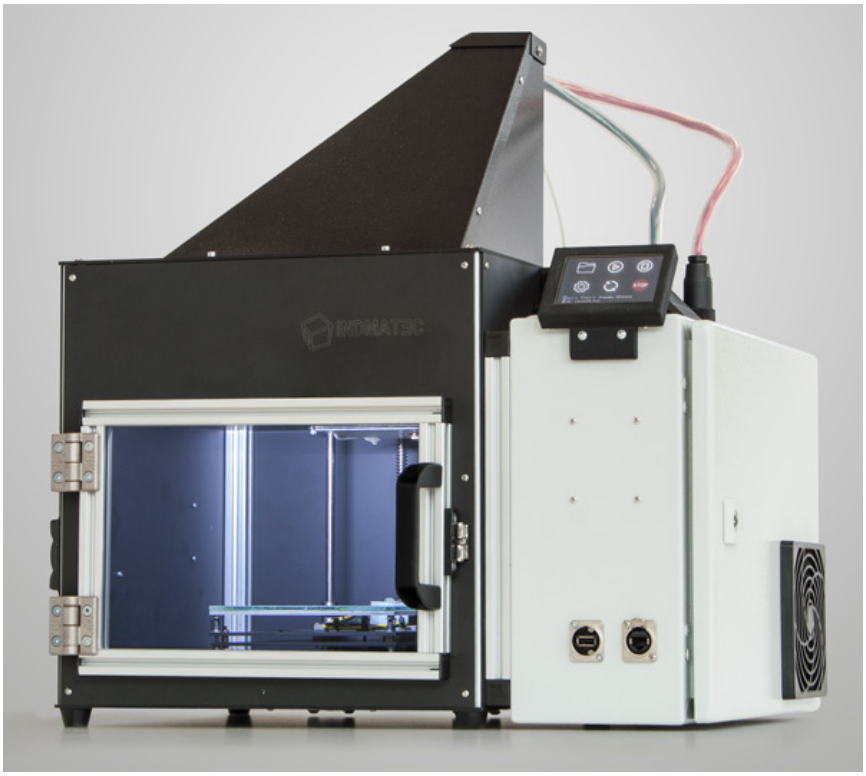

Figure 1-3: Apium Industries HPP 155 (Now discontinued and replaced with P 155)

2 MATERIALS AND METHODS

2.1 Design and Fabrication of samples

All 3D printed samples in this study were made on the Apium HPP 155 PEEK printer. This is a very new process and printer and there were many issues in optimally using the printer. During the course of study the printer had to be replaced but all samples in this study are printed on the new printer.

The biggest issue that was faced in printing samples was warping. Because PEEK is printed at such high temperatures and then cooled, the heat gradient in samples and localized stress concentration caused the samples to warp and come off the plate. This problem was overcome by changing the printing speed, optimizing layer height, nozzle temperature and placement of thermocouple sensing the build space temperature.

A few other issues that were faced and optimized during the course of this study are displacement in layers while printing and delamination of parts during cool-down.

2.1.1 Bone plate manufacturing

All simulated bone plate samples were designed according to specifications described in ASTM F382. Each sample was 3.2 mm in thickness, 114 mm in length and 12.8 mm in depth. The six holes for the bone screws are 4.5 mm in diameter. A spherical countersink of 3mm radius and 1.06mm depth was provided on all samples to ensure proper meshing with the 3.5 mm cortical screw which has the same radius of the face in contact. Samples with different depths of countersink, 1.6 mm, 1.06 mm and 0.53 mm were made. The sample with 0.53 mm countersink had very less penetration of the screw head and the raised screw head is an undesirable factor in a bone plate system, whereas the sample with 1.5 mm countersink was weakened at the holes due to excessive penetration of the screw head and failed more easily at the holes as compared to the 1.06 mm countersink.

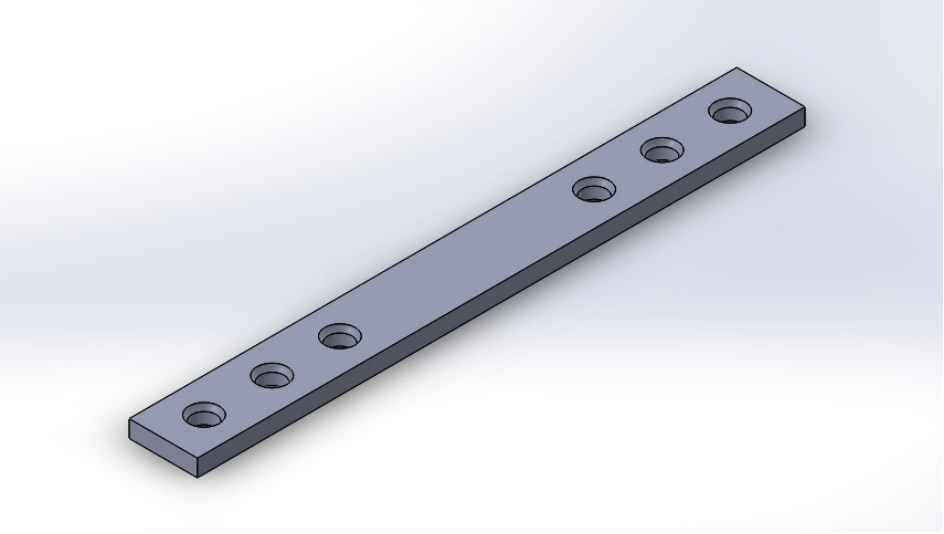

Figure 2-1: CAD model of the bone plate used in study

Samples manufactured by additive manufacturing were printed in two orientations, XZ and XY. Samples were made of 50% and 100% infill in both of those orientations. The infill was

rectilinear with alternate 0° and 90° layers w.r.t. the X axis. All samples had 3 outer shells. This means that all outer surfaces had 3 solid layers of material irrespective of infill percentage. Because of this the 50% infill samples had larger differences in the actual amount of material they contained. 100% and 50% infill samples in the XY orientation contained approximately 4.9 cm^3 and 3.8 cm^3 of material respectively whereas 100% and 50% infill samples in the XZ orientation contained 4.8 cm^3 and 4.5 cm^3 of material respectively.

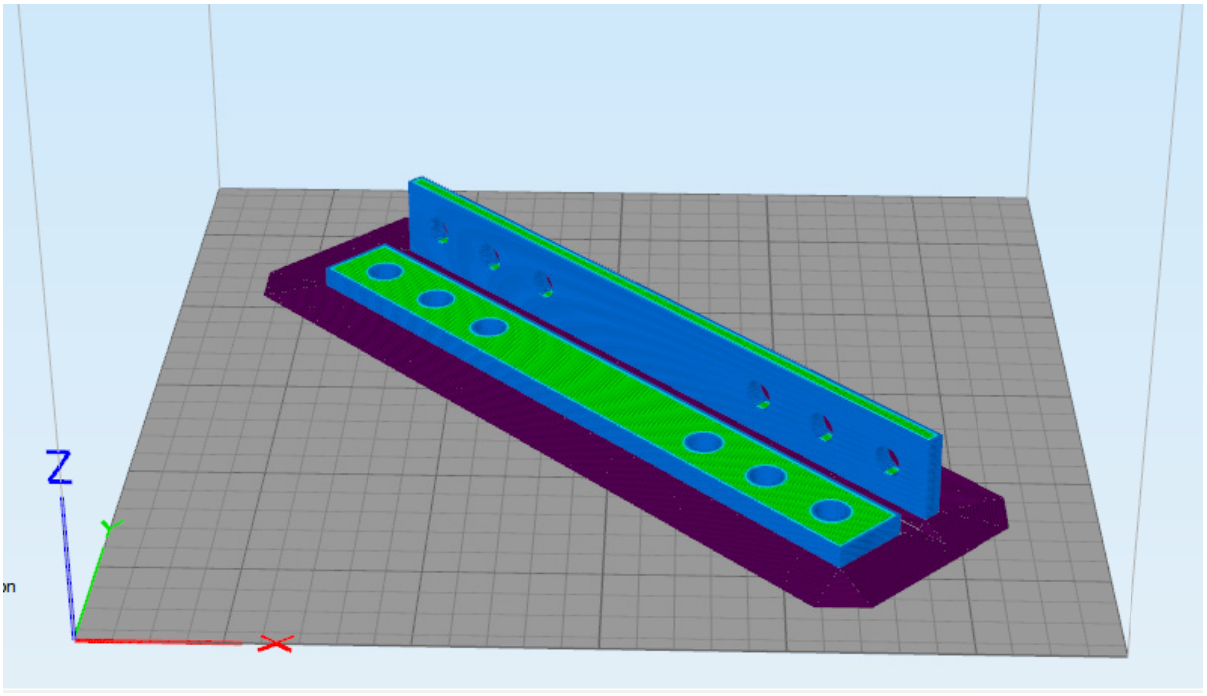

Figure 2-2: Figure showing the bone plates being prepared for printing in XY and XZ planes

The control samples were machined from a 3.2mm PEEK sheet and the spherical countersink for the cortical screws were machined using ball end mills later in the experiment for the fatigue tests.

Figure 2-3: Machined bone plate sample made from sheet of PEEK

2.2 Design and fabrication of simulated bones and cortical screws

The samples were mounted on simulated bone structures made from polyacetal Delrin®. These simulated bones were previously fabricated for another study [17] and were reused for this study. They were fabricated using CNC milling. Flat surfaces were machined on opposite sides of the hollow cylinder bone segments to accommodate the flat bone plate. Holes were drilled and tapped to accept the bone screws, and grooves were added to the bottom transversely to aid in locating and placing the segments on the on cylindrical supports.

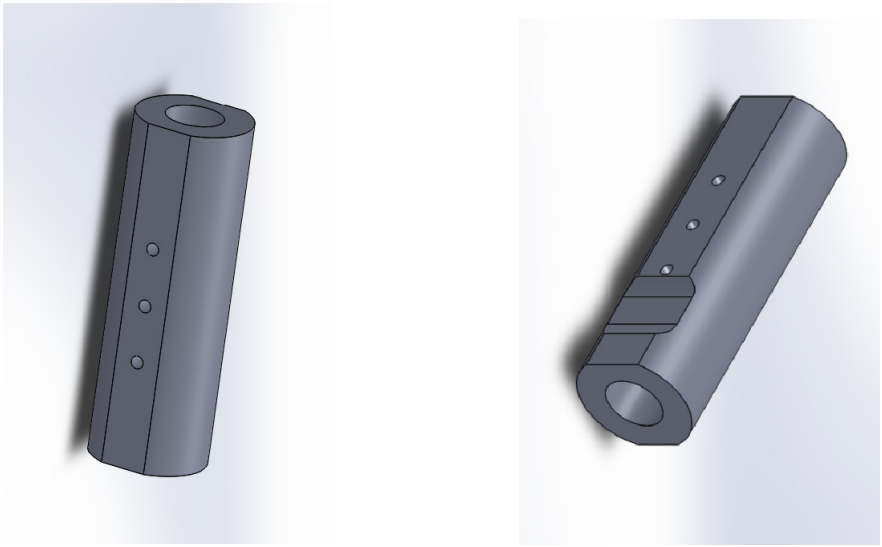

Figure 2-4: Top of simulated Delrin bone with holes for attaching bone plate (Left), Groove on the bottom end to help locate on the testing fixture (Right)

The bone plates were mounted onto the simulated bone using 3.5 mm cortical screws and the flat head cortical screw for the initial strength tests. The reason for this is described in the next part of this section. The screws were designed in SolidWorks but due to issues with the manufacturing of them, screws used in this study were commercial titanium cortical screws used in a previous study.

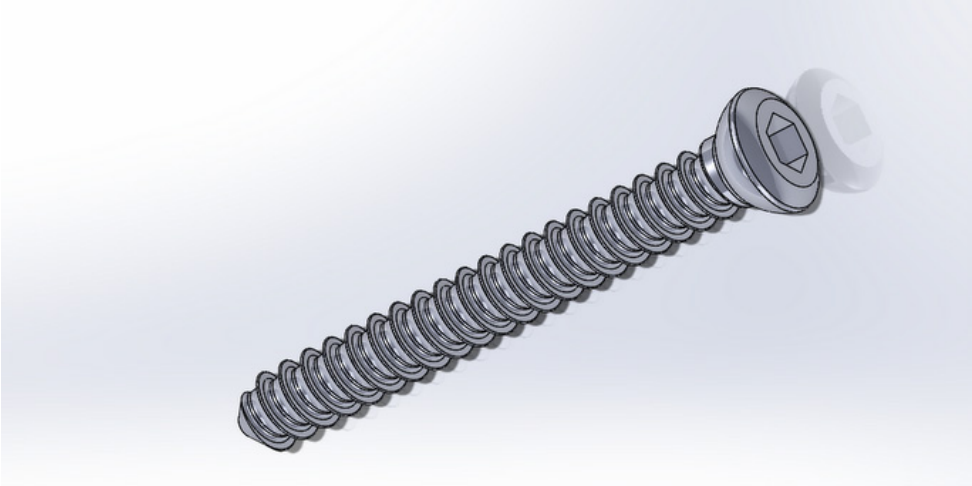

Figure 2-5: CAD model of designed 3.5 mm cortical screw

Figure 2-6: CAD model of designed 3.5 mm cortical screw with flat head

Reason for using flat head cortical screws in strength testing

The samples were assembled and put together using two simulated bone segments to which a bone plate was attached by a total of 6 cortical bone screws. This will be referred to as the

construct. It was found that the samples printed in the XZ plane failed by delaminating upon tightening of cortical screws. Samples of 50% and 100% infill, printed in the XZ plane were tested using a calibrated torque wrench to find out the torque they fail at.

All XZ samples with 50% infill delaminated at torque values less than 0.34 Nm. Apart from two samples, which failed at 0.4 Nm and 0.45 Nm all other samples failed at less than 0.34 Nm. The samples printed in the XY plane were intact at 1.29 Nm, which was the maximum range of the torque wrench.

All initial strength testing was done using flat head cortical screws so that we can compare stiffness values over all different types of samples and establish a baseline for stiffness value. All the samples were tightened to a torque of approximately 0.8 Nm for testing.

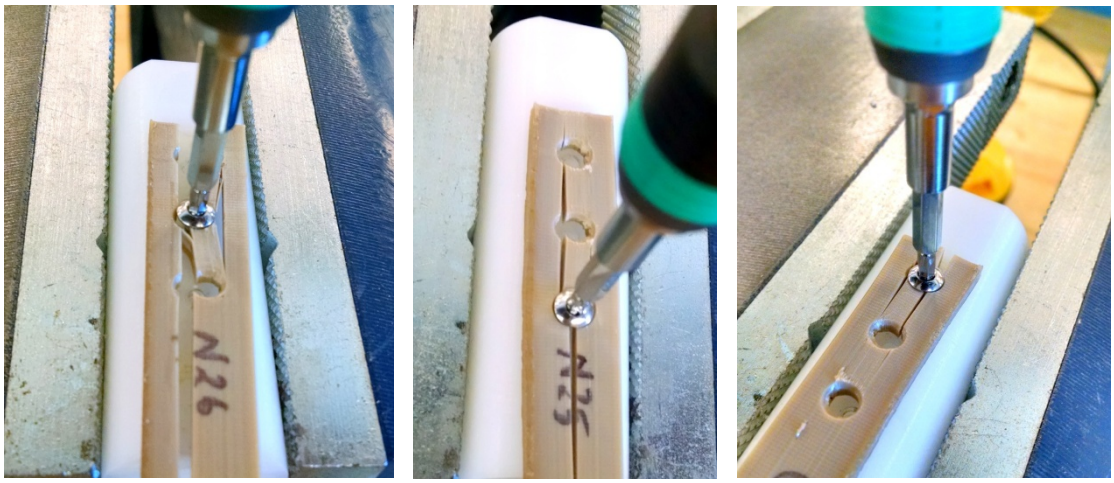

Figure 2-7: Samples printed in XZ plane delaminating under application of torque

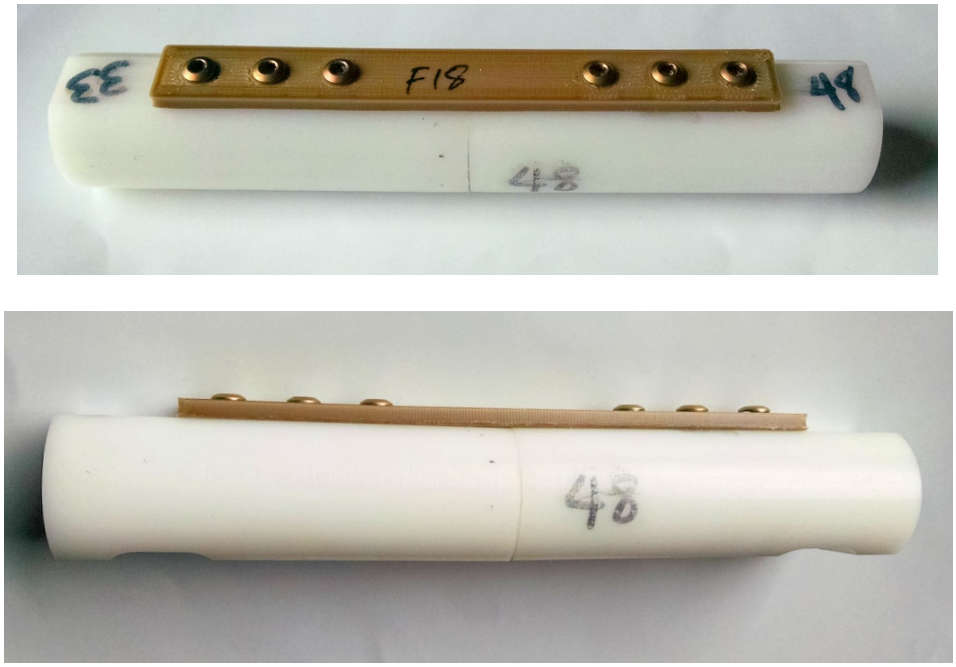

Figure 2-8: Photos of assembled constructs

2.3 Mechanical Testing Procedure

2.3.1 Strength testing

Strength testing was performed according to ASTM F382 which is typically for metallic bone plates but was extended to polymeric bone plates for this study. A four point bending procedure was followed for this test. The samples were tested on the ATS universal testing machine. The support fixture had a major spacing of 127 mm and the load head attached to the crosshead and to a 250 lb load cell has a spacing of 25.4 mm between the loading points. A preload of less than 2 N was applied to the sample before the beginning of the test. The loading rate was set to 6.35mm/min. The stopping condition was failure of the sample or

displacement of the sample exceeded 18.2 mm after which the sample was no longer in the intended position on the supports. Load and displacement data were collected for comparison.

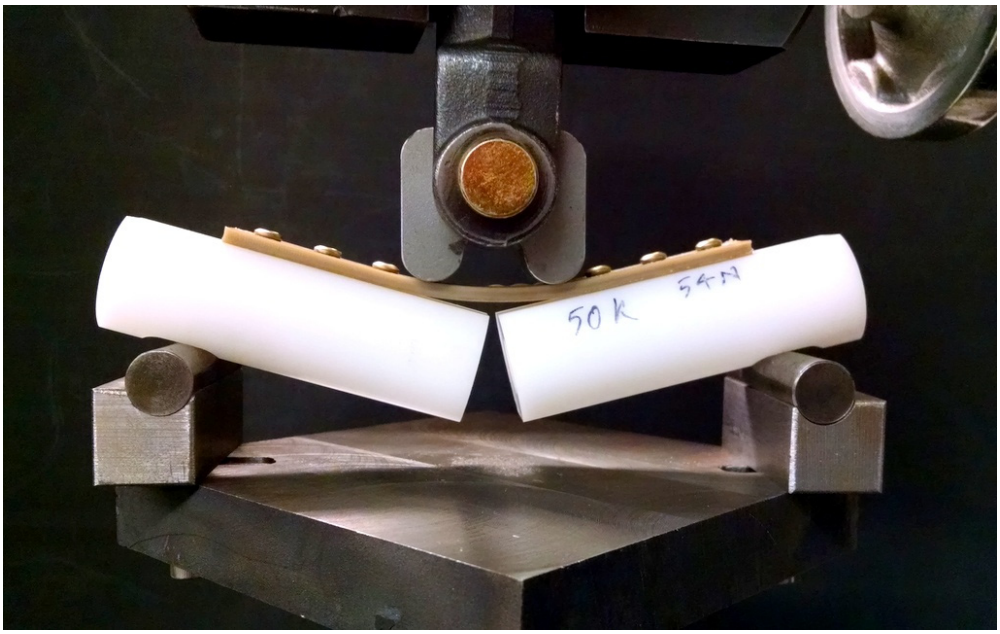

Figure 2-9: Four point bending test setup for strength testing of constructs

Due to the delamination problems faced when applying torque on the cortical screws on samples printed in the XZ plane, strength tests were carried out with screws with flat heads but same thread profile. The samples printed for the strength tests did not have a spherical countersink.

2.3.2 Fatigue testing

Fatigue testing of samples was done using the method prescribed in ASTM F382 on an Instron dynamic testing machine. The setup was exactly the same as the one used for strength test. The test was performed on an Instron testing machine at a frequency of 6 Hz. Samples were put under a preload of 3 N to ensure that the loading head does not rotate during the test.

Due to sub-optimal PID tuning of the load control method for the machine displacement control was used. As the samples have minimal yielding before failure this method of testing gave accurate results. One set of 5 samples was loaded to 54 N and was run for 50,000 cycles. Another set of 5 samples was loaded to at 62 N and was run to 100,000 cycles. This load was calculated as 80% of the minimum and maximum load that 100% infill samples failed at in strength testing. To check additional durability one sample was run at a load of 54 N to 350,000 cycles. All control samples were tested at 62 N and run for 200,000 cycles.

The displacement corresponding to necessary load was obtained for every sample and accordingly the amplitude of the sine wave for the displacement of the loading head was calculated.

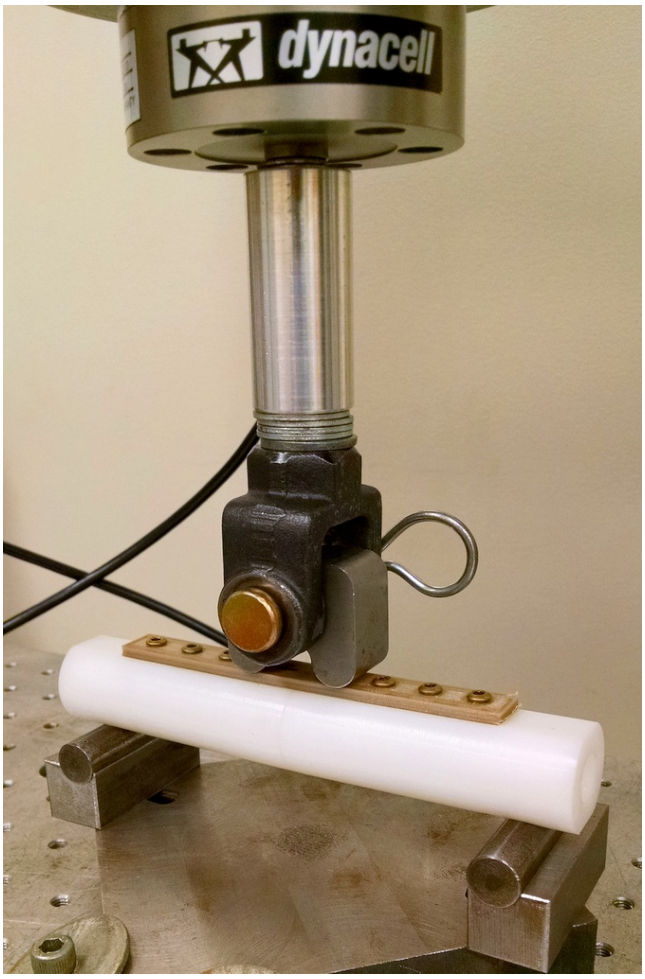

Figure 2-10: Test setup for fatigue testing on the Instron testing machine

Residual strength was checked for every sample after the fatigue tests by running them through a strength test.

3 RESULTS AND DISCUSSION

3.1 Nomenclature of samples

Samples printed for strength testing were labeled with the prefix 'N' followed by a number with s suffix indicating the infill percentage and build orientation. Samples printed in the XY plane were denoted by 'H' and samples printed in the XZ plane were denoted by 'V'. For example a sample printed at the 23rd number with 100% infill in the XY plane would be denoted as N23 100H. All samples with prefix 'N' did not have a spherical countersink on the holes for screws.

Samples printed for fatigue testing were labeled with the prefix 'F' followed by a number. All samples printed for fatigue testing had 100% infill for the reasons described in the following part of this chapter.

All control samples which were machined from a 3.2 mm sheet of PEEK were labeled with the prefix 'C'.

3.2 Strength tests

Strength tests were performed to check the maximum strength of the constructs and their stiffness. Graphs for the load vs. displacement graph are given on the following pages. The place where the load drastically falls is the region of failure. The place where load rapidly increases is the displacement where the line of contact of the outer supports is at the edge of the groove cut into the simulated bone (as shown in Figure 3-1).

All strength tests as reported before were performed using the flat headed cortical screw to attach the bone plate to the simulated bone because the samples printed in the XZ direction delaminated upon torque application.

Figure 3-1: Line of contact of support roller at the edge of the groove

The graph below (Figure 3-2) shows the difference in stiffness between the 100% and 50% infill samples printed in the XY plane. It is important to note here that all 100H samples

failed whereas only half of the 50H samples failed. The stiffness of the 50H samples is much lower than that of the 100H samples which can be attributed to the amount of material they contain (4.9 cm^3 for 100H and 3.8 cm^3 for 50H).

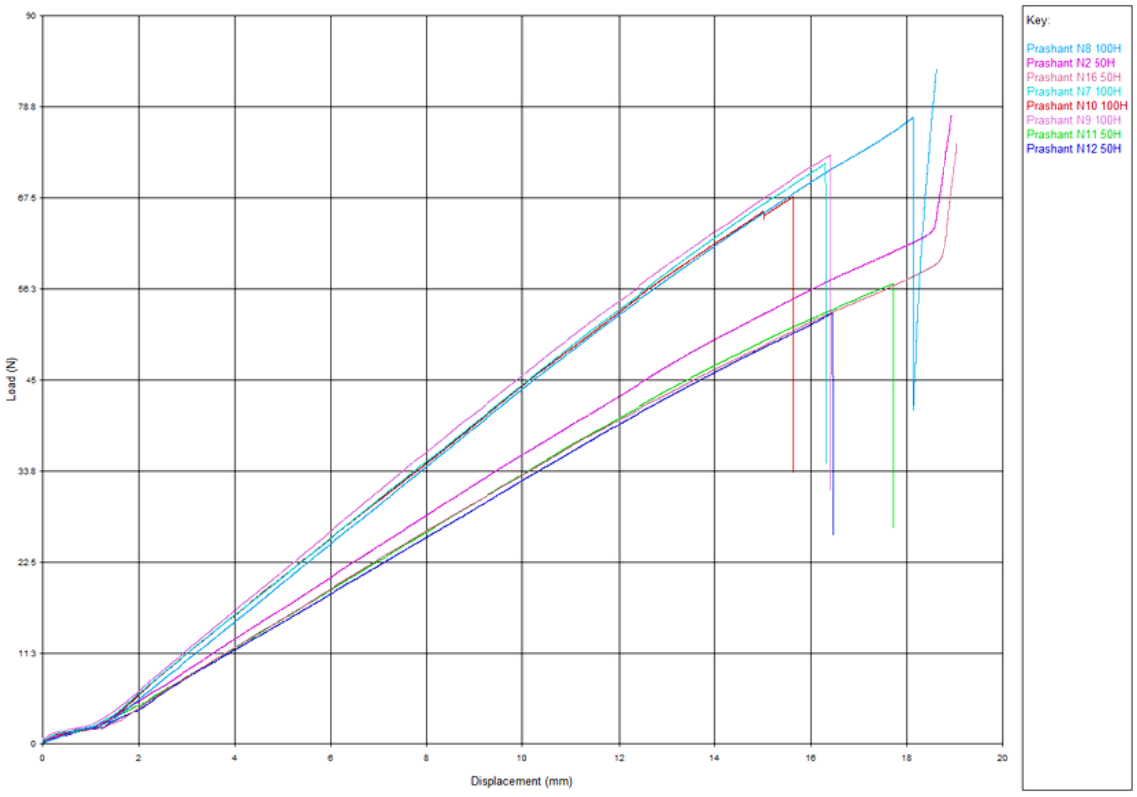

Figure 3-2: Load vs. displacement graphs for 100H and 50H samples

The graph below (Figure 3-3) shows the difference in stiffness between the 100% and 50% infill samples printed in the XZ plane. Only two 50V samples failed in this test. The stiffness of the 100V and 50V samples is comparable. This is because of the orientation of printing. Because the samples were printed in the XZ plane the volume of sample that qualified for an infill was low. That is why the material used to print 100V and 50V samples is almost the same (4.8 cm^3 for 100V and 4.5 cm^3 for 50V). The 100V and 50V samples failed when applying torque using the cortical screws. This is why these were both declared unfit as an actual bone plate and were thus excluded from the fatigue testing study.

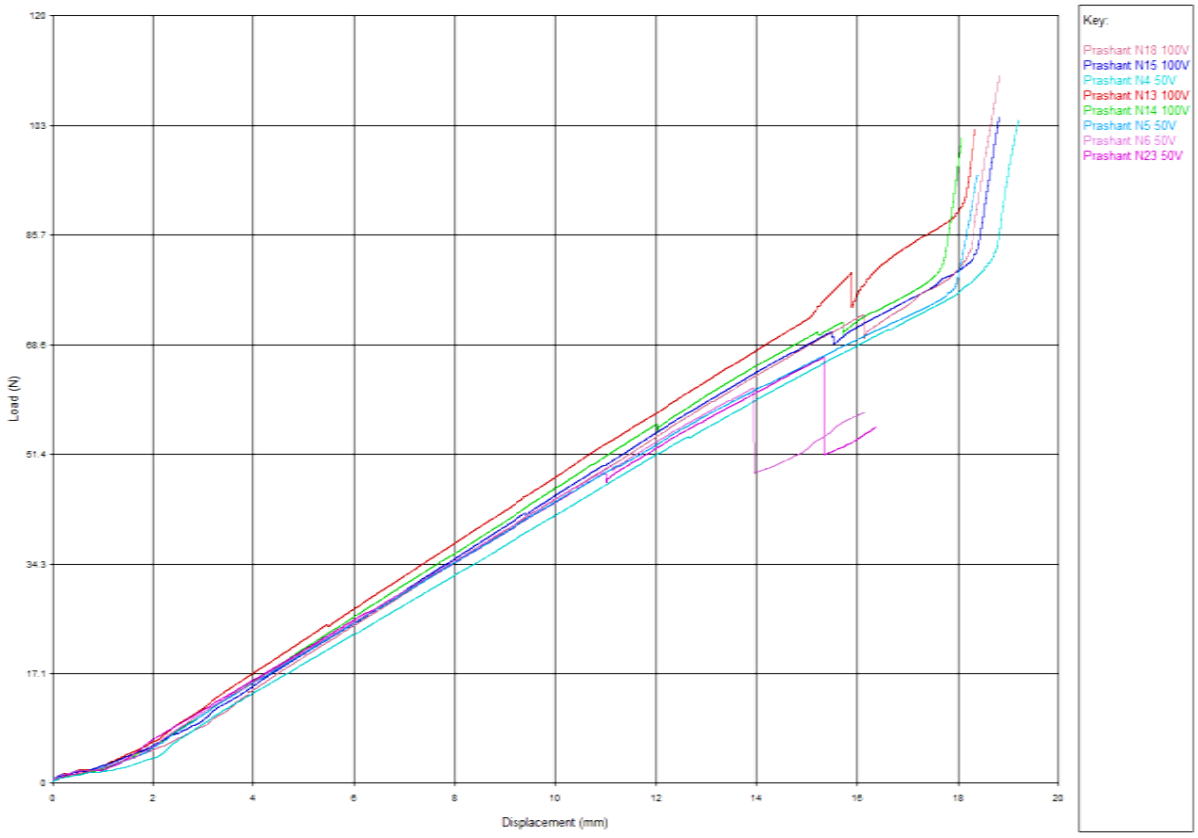

Figure 3-3: Load vs. displacement graphs for 100V and 50V samples

The graph below (Figure 3-4) shows the difference in stiffness between the control samples and 100% infill samples in XY and XZ plane. While the stiffness of the control samples is slightly higher, the stiffness of 100H and 100V samples is comparable.

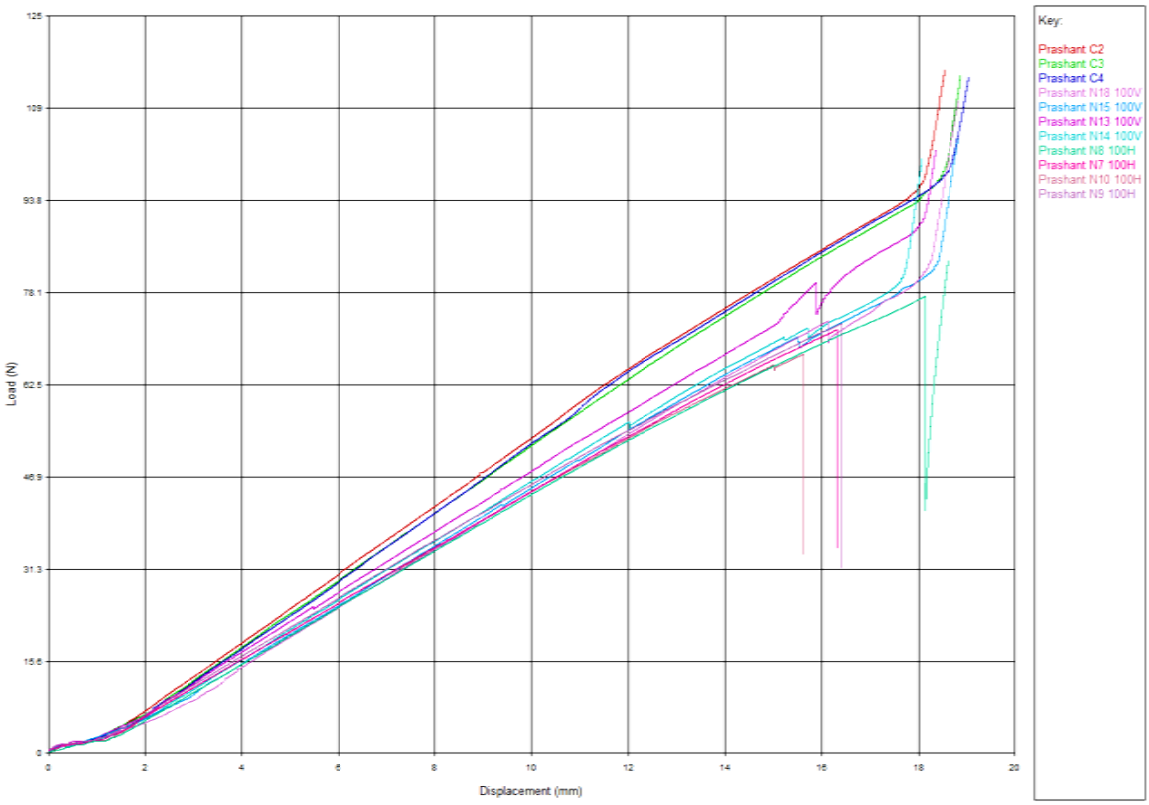

Figure 3-4: Load vs. displacement graphs for Control, 100H and 100V samples

Table 1 shows the stiffness values of all the samples tested in the four point bending test. Due to very low standard deviation only four samples were tested. All stiffness was calculated using the displacement caused in the loading span of 20 – 40N.

The stiffness of the 50H samples is much lesser than the control and 100H. Due to this difference in stiffness and variation from control the 50H samples were excluded from the fatigue testing study. Figure 3-5 pictorially represents the values given in the table.

Table 1 - Stiffness values of strength tested samples

Stiffness values of strength tested samples					
Sample type	100 H	100V	50 H	50 V	Control
Sample 1	4.744	5.083	3.758	4.64	5.88
Sample 2	4.777	4.982	3.557	4.735	5.793
Sample 3	4.903	5.03	3.503	4.779	5.645
Sample 4	4.665	4.935	3.475	4.559	5.785
Average	4.77225	5.0075	3.57325	4.67825	5.77575
Standard Deviation	0.099027	0.063543	0.127782	0.098412	0.097206

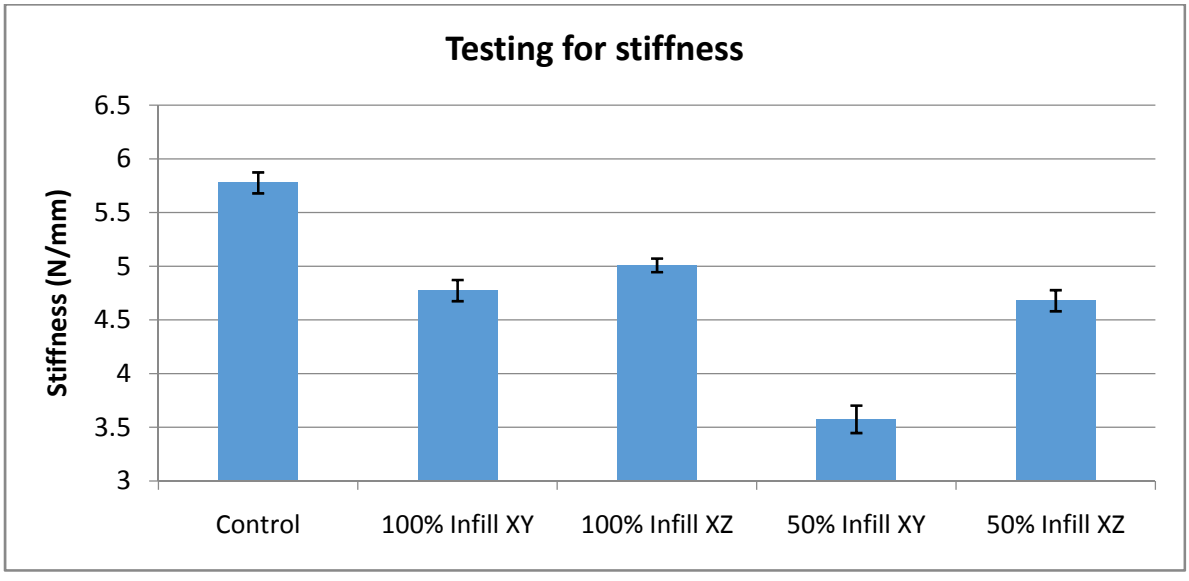

Figure 3-5: Comparison of average stiffness of samples tested in strength testing

3.3 Fatigue test and residual strength test

After the results of the strength testing only one type of sample (100H) was tested for fatigue life along with the control samples. All intact samples were tested for residual strength test after the fatigue test cycle was completed.

Table 2: Test matrix of samples qualified for fatigue testing

	100% infill XY plane	100% infill XZ plane	50% infill XY plane	50% infill XZ plane
Stiffness in strength test	Comparable to control	Comparable to control	Very less compared to control	Comparable to control
Withstanding torque using cortical screw	Passed	Failed	Passed	Failed
Final outcome for fatigue testing	**OK**	**Eliminated**	**Eliminated**	**Eliminated**

- The loading values for the fatigue tests were 80% of the minimum and maximum value of load at which the 100H samples failed. These values are 54 N and 62 N respectively.
- First set of 5 samples was run for 50,000 cycles at a load of 54 N. Because no sample showed any kind of damage the 5th sample was run till 350,000 cycles at the same load.
- Because no sign of wear showed on the 5th samples the next set of 5 samples were run for 100,000 cycles each at a load of 62 N. The 5th sample in this set gradually started cracking at 80,000 cycles and failed in the next 1500 cycles.

- The set of 4 control samples were run for 200,000 cycles at a load of 62 N and showed no signs of damage.

Strength tests were performed on all the fatigue tested samples to find out the residual stiffness.

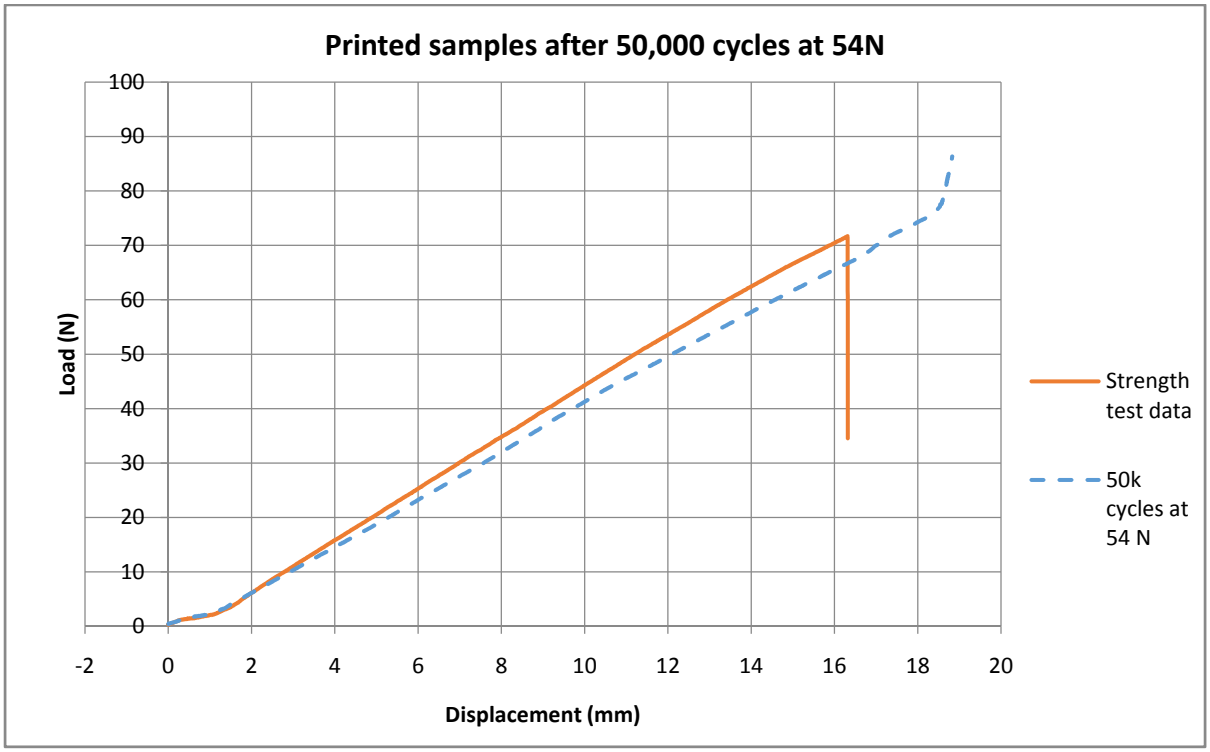

Figure 3-6: Comparison of average residual strength between printed samples after 50,000 cycles at 54N and initial strength test data

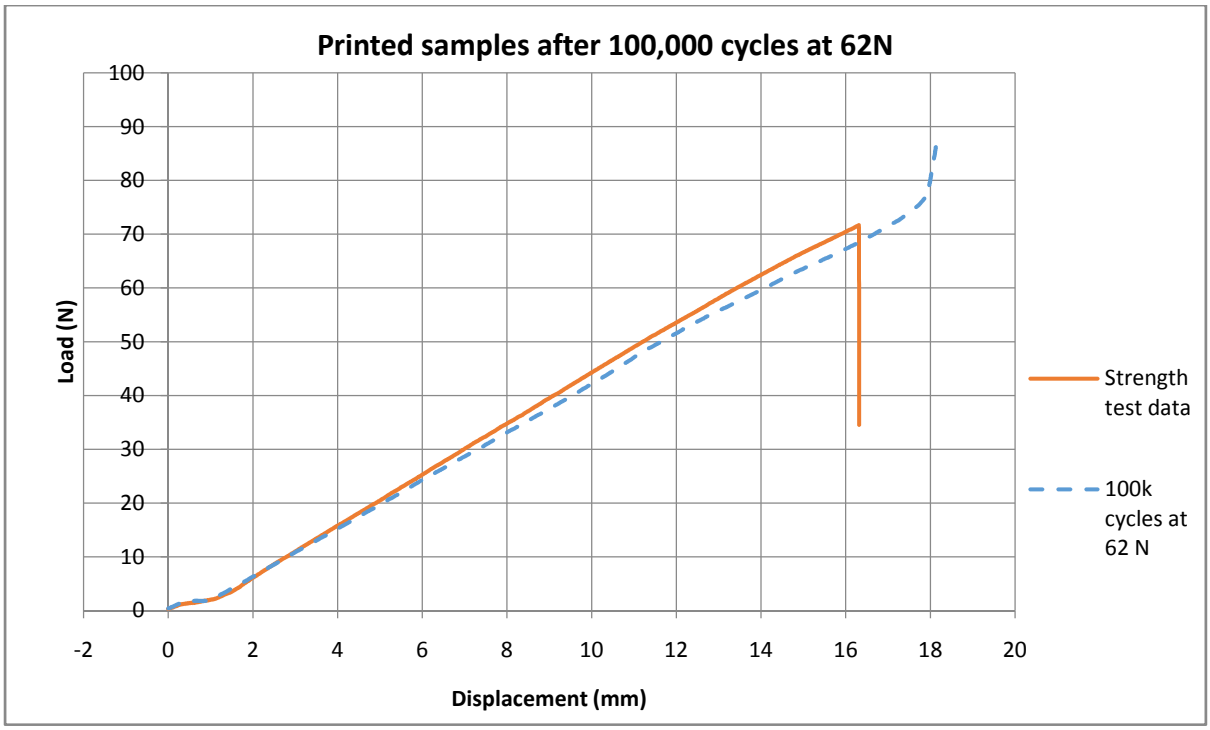

Figure 3-7: Comparison of average residual strength between printed samples after 100,000 cycles at 62N and initial strength test data

One sample from the first set that ran for 50,000 cycles failed during the strength test but had the same stiffness as the other samples from the set. As expected there is a reduction in stiffness in the printed samples but we can see from the figures above that this reduction is not significant.

Table 3: Residual stiffness after fatigue in printed samples

Residual stiffness after fatigue in printed samples			
Sample type	100 H	50k cycles at 54 N	100k cycles at 62 N
Sample 1	4.744	4.444	4.588
Sample 2	4.777	4.471	4.688
Sample 3	4.903	4.669	4.378
Sample 4	4.665	4.502	4.44
Average	4.77225	4.5215	4.5235
Standard Deviation	0.099027	0.101148	0.140669

When control samples were tested for residual stiffness it was found that the stiffness of the samples had actually increased after fatigue testing. The only difference in the samples here is the change in screw from a flat head cortical screw in the initial strength testing to a cortical screw in the fatigue and residual strength testing. This is further investigated in the next section

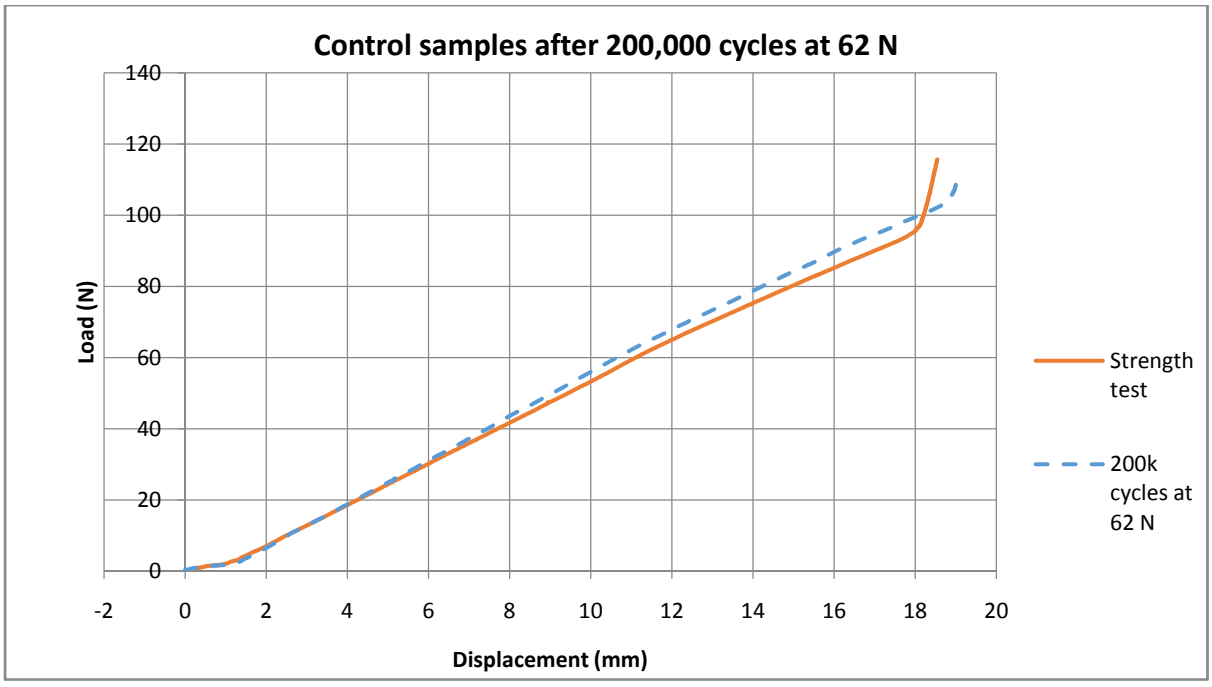

Figure 3-8: Comparison of average residual strength between control samples after 200,000 cycles at 62N and initial strength test data

Table 4: Residual stiffness after fatigue for control samples

Residual stiffness after fatigue for control samples		
Sample type	Control	200k tested at 62 N
Sample 1	5.88	5.939
Sample 2	5.793	6.2
Sample 3	5.645	6.005
Sample 4	5.785	6.376
Average	5.77575	6.13
Standard Deviation	0.097206	0.197924

3.4 Discussion

A new set of control samples was tested for stiffness but this time cortical screws used to secure the bone plate to the simulated bones. The stiffness values were compared to initial values that were determined using flat headed cortical screws. From the Figure 3-9 below we see that control samples are stiffer when secured using cortical screws.

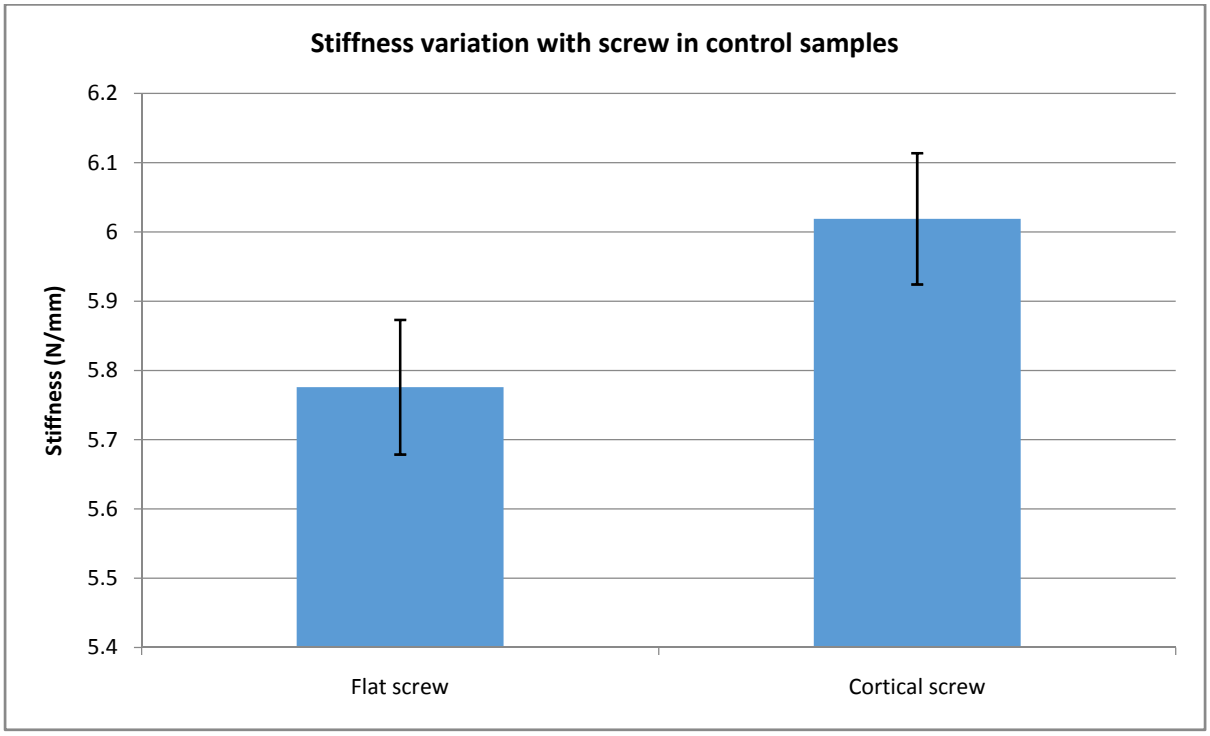

Figure 3-9: Stiffness variation with screw in control samples

Similarly a new set of four 100% infill XY orientation samples were printed and tested for stiffness with cortical screws. This time it was found that the stiffness decreased when cortical screws were used to secure the bone plate (Figure 3-10).

Table 5: Stiffness variation with screw in Printed samples

Stiffness variation with screw in Printed samples		
Sample type	Cortical Screw 100 H	Flat head screw 100 H
Sample 1	4.508	4.744
Sample 2	4.485	4.777
Sample 3	4.615	4.903
Sample 4	4.473	4.665
Average	4.52025	4.77225
Standard Deviation	0.064814	0.099027

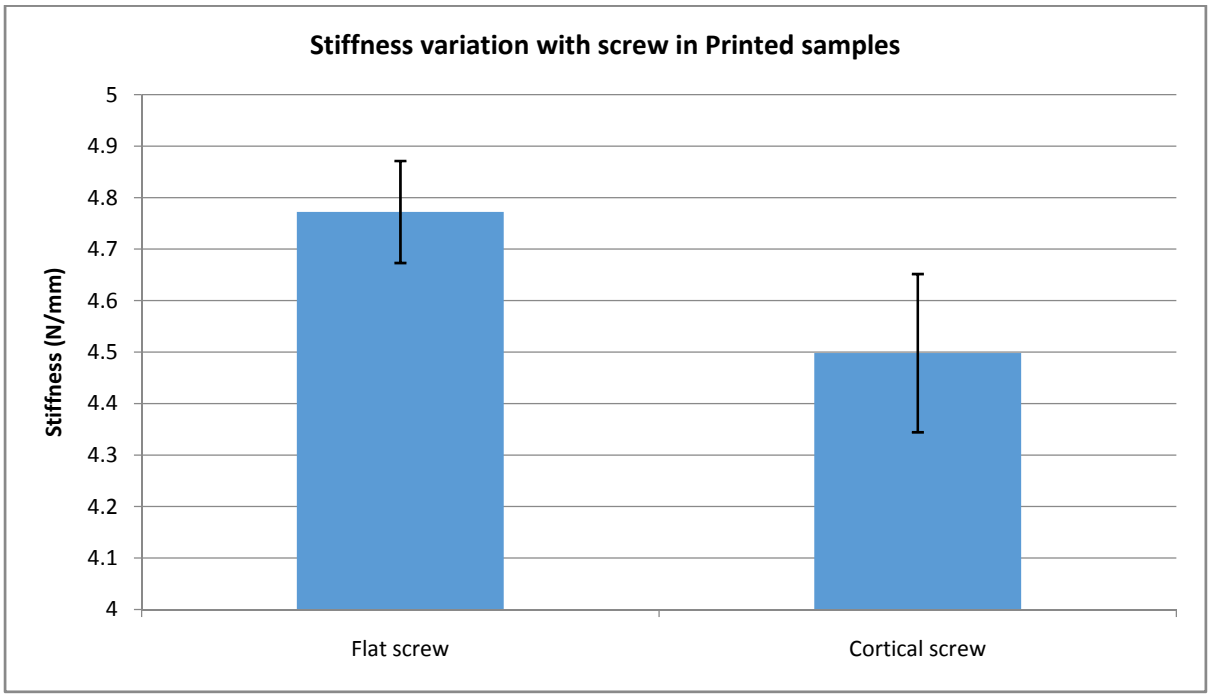

Figure 3-10: Stiffness variation with screw in Printed samples

From figure 3-11 we can see that the values of stiffness in control samples increase slightly with the use of cortical screws and decrease in the case of printed samples. It is interesting to note that none of the printed samples tested for stiffness with the cortical screw failed.

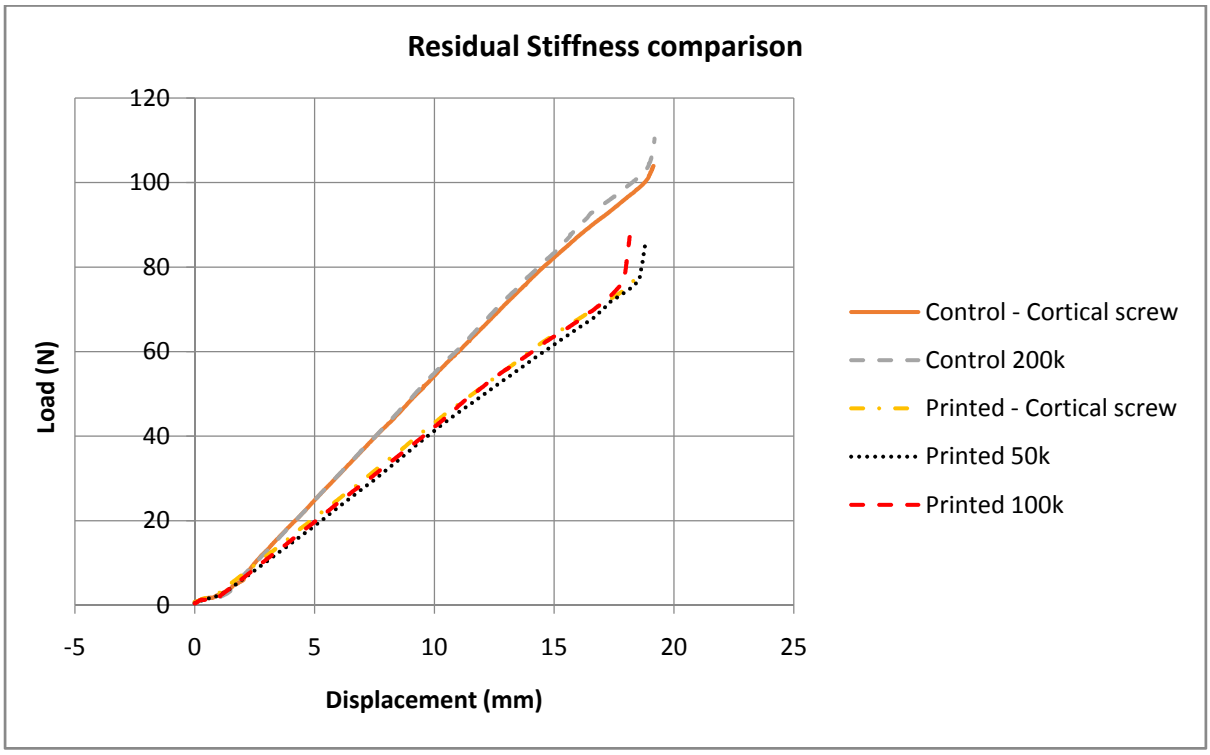

Figure 3-11: Residual Stiffness comparison

The possible reasons for this phenomenon are listed below.

- The clamping force is distributed in the body of the sample in case of a spherical counter-sink along the length instead of it being localized to a small area perpendicular to thickness in non counter-sink sample as shown in the figure 3-12 below.

- *Control Sample:* The clamping force being distributed along the length in case of a sample with a counter-sink, introduces pre-stress in the sample which causes an increase in its stiffness.

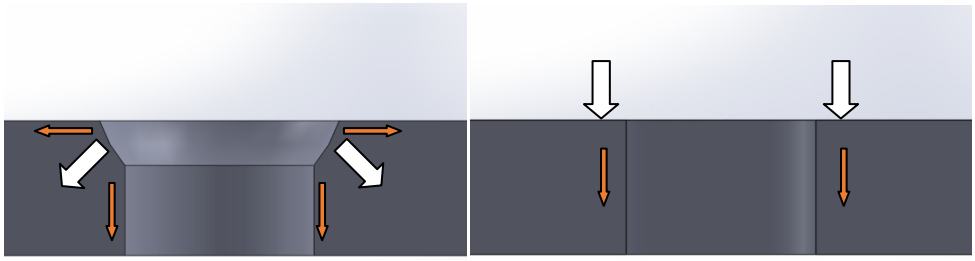

Figure 3-12: The white arrows show the force exerted by the screw on the plate and the orange arrows show how that clamping force is distributed through the bone plate. The plate to the left has a spherical countersink and is fixed by a cortical screw and the plate

- *Printed Sample:* In case of a sample without countersink the stress concentration are high near the screw holes which cause them to fail during strength testing. But in the case of a sample with a counter-sink the stiffness does not increase. This might be due to the fact that the sample is printed in layers and has outer shells which prevent the sample from developing a pre-stress due to the compliance present in the interface. This is shown in the figures below.

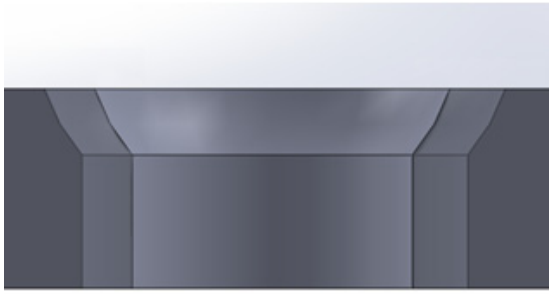

Figure 3-13: Printing path around the hole is the intermediate shade of gray.

Figure 3-14: Magnified view of an intermediate layer of the sample showing interface between infill and outer shell

- The 3D printer has been under constant improvement with correspondence with the manufacturer. As the temperature is of the build plate and nozzle is tweaked the crystallinity percentage of the sample might have changed leading to a change in the mechanical properties [16].

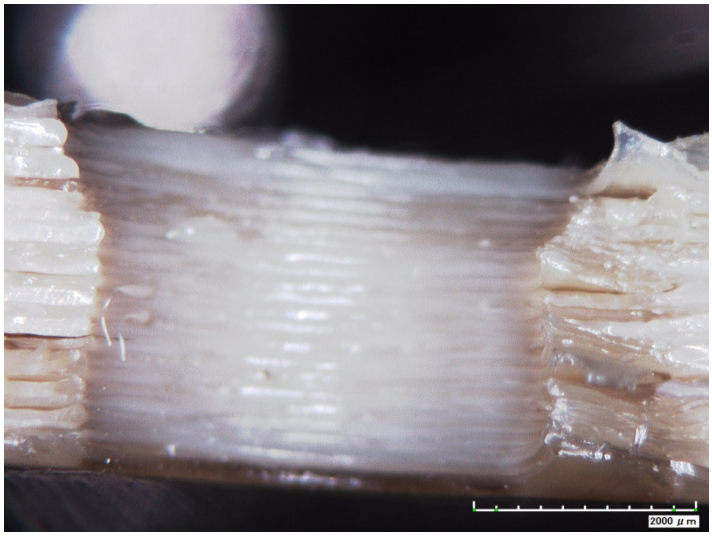

Figure 3-15: Magnified cross section of hole in printed sample showing different amounts of crystallinity in layers seen as light and darker PEEK

4 CONCLUSION AND FUTURE WORK

4.1 Conclusion

In this study the mechanical properties of 3D printed PEEK and machined compression molded PEEK as bone plates were studied in strength testing where they were subjected to a 4 point bending load and fatigue testing. These were to simulate the loading conditions that a bone plate might be subjected to in real life. Out of the four types of samples printed only one sample qualified to be used as a bone plate and was run in fatigue testing. The stiffness of this sample was around 17% lower than the control sample.

The reduction in stiffness due to fatigue testing up to 100,000 cycles at a load 80% of the breaking load of the strength tested printed samples was minimal. It was later discovered that the samples did not break when the type of screws used to secure them to the simulated bone were changed. This can also be attributed to the change in crystallinity of the samples as the 3D printer was improved as the study was performed.

The defects and voids in the printed samples which gave rise to variability in the longevity of the samples under fatigue testing but caused very little variance in the strength testing. Due to this variability and the lack of control required over the temperature in the 3D printing process and the Apium HPP 155 printer 3D printed PEEK samples do not match up to the standards of machined PEEK for use as a bone plate.

4.2 Recommendations for future research

- Due to time constraint the samples could not be tested in fatigue to failure or to the extent to support its function as a bone plate (approximately 1.4 x 10^6 cycles). Destructive cross section analysis of samples should be done at regular cycle intervals to note the internal and invisible damage and delamination happening between the layers in the highest stress zone area.
- Optimization of the 3D printer should be avoided if possible during the course of the study to achieve increased consistency in the printed samples.
- Study of non-loadbearing implants such as facial and cranial implants can be done.
- Mechanical properties of 3D printed PEEK parts coated with titanium or a bioactive/osteoconductive material like Hydroxyapatite can be studied.
- The PEEK printer used in this study was first of its kind and so had a lot of problems improvable defects in it. With time, as better printers become available samples can be made with lesser variability and better test results can be obtained.
- Use of biodegradable materials such as Polyglycolic acid and polycapolactone in juncture with PEEK can be investigated to improve biomedical implant quality.
- The results gotten from this study can be analyzed by FEA assuming the printed PEEK to be a composite material.

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