Nondestructive Ultrasonic Quality Testing of Endovascular Devices

Robert Earl

Department of Mechanical Engineering

McGill University, Montreal

April 2021

A thesis submitted to McGill University in partial fulfillment of the requirements of the degree of Master of Engineering

©Robert Earl, 2021
ACKNOWLEDGEMENTS

I would like to acknowledge and thank the following for their numerous and invaluable contributions:

- **Professor Richard L. Leask** for his guidance, support, and encouragement throughout the project and pandemic.
- **Professor Rosaire Mongrain** for his motivation, excitement, and passion that helped see the value and possibilities even in the early stages of the project.
- **Professor Paul Fromme** for his guidance on non-destructive evaluation using ultrasound, his preliminary proof of concept work, and helping to interpret the results throughout the project.
- **Dr. Amin Jafari Sojahrood** for his assistance with the experimental setup, and preliminary proof of concept work.

This work was supported through [NSERC Engage (EGP #533972-18)] and the Implementation of Innovation in Industry in Quebec [Triple IQ Program] in collaboration with [Agile MV], the industry sponsor.
# TABLE OF CONTENTS

Acknowledgements ........................................................................................................ i

Table of Contents ........................................................................................................... ii

List of Figures ............................................................................................................... iv

List of Tables ................................................................................................................ viii

ABSTRACT .................................................................................................................. ix

ABRÉGÉ ......................................................................................................................... x

List of Abbreviations .................................................................................................... xi

1. Introduction and Background .................................................................................. 1

   1.1. Introduction ........................................................................................................ 1

   1.2. Objectives of Research .................................................................................... 3

2. Current Literature ..................................................................................................... 5

   2.1. Non-Destructive Evaluation ........................................................................... 5

   2.2. Medical Device Polymers .............................................................................. 8

   2.3. Types of Faults ............................................................................................... 12

   2.4. Ultrasonic Testing ........................................................................................... 14

      2.4.1. Operation Principles .............................................................................. 14

      2.4.2. Transducers ............................................................................................ 18

      2.4.3. Resolution ................................................................................................ 19

3. METHODS ............................................................................................................ 22

   3.1. Sample Creation .............................................................................................. 22

   3.2. Ultrasound Setup Design ............................................................................. 24

   3.3. Setup Verification – Normal Incidence US Imaging ........................................ 26

   3.4. Micro-CT Weld Inspection ............................................................................ 28

   3.5. Weld Inspection - Angled Beam Imaging ....................................................... 31

   3.6. Mechanical Testing ......................................................................................... 33
LIST OF FIGURES

Figure 1: Process Validation Decision Tree, adapted from [4]. .................................................. 2
Figure 2: Balloon catheter example (distal end), weld locations indicated by orange arrows. ..... 3
Figure 3: Polymer Classifications, adapted from [1]................................................................. 10
Figure 4: Thermoplastic classification, adapted from [1].......................................................... 10
Figure 5: Rendering of example weld faults, adapted from [30] ............................................ 13
Figure 6: Snell’s Law representation adapted from [37]. Material impedance difference ($Z_a$ and $Z_b$), incident wave angle ($\theta_i$), reflected wave angle ($\theta_r$), transmitted wave angle ($\theta_t$)............. 16
Figure 7: Example single element piston transducer (Olympus 15MHz Immersion Transducer V319-SU) .................................................................................................................. 18
Figure 8: Phased array beam forming, transducer elements can be selectively activated to maintain beam width while scanning, adapted from [39] ......................................................... 19
Figure 9: Idealized Axial Resolution in PVC............................................................................. 20
Figure 10: Example Welded Sample .......................................................................................... 22
Figure 11: (A) Hot air welding of samples using inner support mandrel and outer FEP heat shrink sleeve. (B) Representation of sample creation................................................................. 23
Figure 12: Experimental Setup Angled US Testing ................................................................. 24
Figure 13: Experimental Setup Diagram ................................................................................... 25
Figure 14: (A) Raw US Signal, top and bottom wall shown (B) Signal processed for top wall thickness measurements. .................................................................................................................. 27
Figure 15: 2D Scan Example of Control Group. ................................................................. 28
Figure 16: 2D Scan Example of Porous Group. ................................................................. 29
Figure 17: 2D Scan Example of Contaminated Group. ......................................................... 29
Figure 18: Reconstructed Cross-Sectional Image of a Sample with the OD and ID wall detection shown in red. .................................................................................................................. 29
Figure 19: Tensile Testing Failure Mode Examples of A) Control B) Porous C) Contaminated .... 33
Figure 20: Wall Thickness Comparison of Micro-CT (range shown in black, average in green) and US (red) ...................................................................................................................................... 36
Figure 21: (A) Ultimate Tensile Strength Across Groups difference in means (P<0.0001). (B) difference in means of elongation at break (P<0.0001). **P<0.01, ***P<0.001 One-way ANOVA with a Bonferroni Post Test. ................................................................. 37

Figure 22: Total Weld Volume Across Sample Groups difference in means (P=0.0064) *P<0.05, **P<0.01, One-way ANOVA with a Bonferroni Post Test................................................................. 38

Figure 23: (A) Void percentage across groups no difference in means (P=0.2077) (B) Debris percentage across groups difference in means (P<0.0001) ***P<0.001 One-way ANOVA with a Bonferroni Post Test. ........................................................................................................................................ 39

Figure 24: Correlation between total inclusion percentage and ultimate tensile strength (not significant, P = 0.0809, r² = 0.19). Cut off (red dotted line) value at 0.16. Separation value of 0.87. .................................................................................................................................................. 40

Figure 25: (A) Correlation between void percentage and ultimate tensile strength (P = 0.076, r² = 0.3087) (B) Correlation between void percentage and elongation at break (P = 0.042, r² = 0.3845) ........................................................................................................................................ 41

Figure 26: (A) Correlation between debris percentage and ultimate tensile strength (P<0.0001, r² = 0.9077) (B) Correlation between debris percentage and elongation at break (P< 0.0001, r² = 0.8930) ........................................................................................................................................ 41

Figure 27: (A) C-scan image of porous sample with time-of-flight estimated wall thickness (B) C-scan image of contaminated sample with time of flight estimated wall thickness .......... 43

Figure 28: Total normalized energy across groups, significant difference in means (P=0.0059) **P<0.01, One-way ANOVA with a Bonferroni Post Test................................................................. 44

Figure 29: Comparison of Micro-CT and US evaluations via total Inclusion Percentage versus Total energy response post bandpass filtering (2.5-30MHz). Contaminated sample 5 (red) energy did not increase with increased total inclusion percentage.................................................. 45

Figure 30: Correlation between total normalized energy response post bandpass filtering (2.5-30MHz) and ultimate tensile strength (P= 0.0101, R² = 0.3869). Control group average 0.14±0.06. Cut off (red dotted line) at 0.33. Separation value of 1.3.................................................. 46

Figure 31: (A) Difference in mean normalized energy across groups with 5-10MHz bandpass filtering (P =0.0044) *P≤0.05, **P<0.01 One-way ANOVA with a Bonferroni Post Test............. 47
Figure 32: (A) Correlation between energy response post bandpass filtering (5-10MHz) versus ultimate tensile strength (P=0.0040, \( r^2 = 0.4576 \)). Control group average 0.57±0.22. Cut off (red dotted line) at 1.24. Separation value of 2.1. (B) Energy response post bandpass filtering (5-10MHz) versus strain at break (P=0.0002, \( r^2 = 0.6312 \)). Control group average 0.57±0.22. Cut off (red dotted line) at 1.24. Separation value of 2.1.

Figure 33: Mean difference across groups maximum normalized single trace energy post 5-15MHz bandpass filtering (P=0.0204) *P≤0.05 One-way ANOVA with a Bonferroni Post Test...

Figure 34: (A) Normalized maximum single trace energy response post bandpass filtering (5-15MHz) versus ultimate tensile strength. No significant correlation. Control group average maximum single trace energy 0.27±0.10. Cut off (red dotted line) at 0.57. Separation value of 0.9. (B) Normalized maximum single trace energy response post bandpass filtering (5-15 MHz) versus elongation at break. Weak correlation (P=0.0295, \( r^2 = 0.2957 \)). Control group average maximum single trace energy 0.27±0.10. Cut off (red dotted line) at 0.57. Separation value of 0.9.

Figure 35: (A) Correlation between normalized energy response of porous and controls post bandpass filtering (5-15MHz) versus ultimate tensile strength (P=0.0025, \( r^2 = 0.6551 \)). Control group average normalized energy 0.38±0.17. Cut off (red dotted line) at 0.88. Separation value of 2.9. (B) Correlation between energy response of porous and controls post bandpass filtering (5-15MHz) versus strain at break (P=0.0002, \( r^2 = 0.7925 \)). Control group average normalized energy 0.38±0.17. Cut off (red dotted line) at 0.88. Separation value of 2.9.

Figure 36: (A) Correlation between normalized energy response of contaminated and controls post bandpass filtering (5-10MHz) and ultimate tensile strength (P=0.0009, \( r^2 = 0.7698 \)). Control group average 0.57±0.22. Cut off (red dotted line) at 1.24. Separation value of 2.1. (B) Correlation between normalized energy response of contaminated and controls post bandpass filtering (5-10MHz) and strain at break (P=0.0022, \( r^2 = 0.7091 \)). Control group average 0.57±0.22. Cut off (red dotted line) at 1.24. Separation value of 2.1.

Figure 37: Rendering of angled beam ultrasound setup. Sample and transducer coupled via water.
Figure 38: Angled Beam US focusing using a half sample. Responses from the half sample was used for normalizing the energy values as resistance was unknown. ................................. 65
Figure 39: Example partial trace used for time of flight wall thickness calculations. ................. 66
Figure 40: Olympus 15MHz transducer frequency spectrum.................................................. 66
Figure 41: Correlation between total inclusion percentage and elongation at break (P = 0.0152, r^2 = 0.3334). Cut off (red dotted line) value at 0.16. Separation value of 0.87................................. 67
LIST OF TABLES

Table 1: Mechanical properties of commodity plastics, adapted from [25-27]. Polyvinyl Chloride (PVC), Linear Low-Density Polyethylene (LLDPE), Low Density Polyethylene (LDPE), High Density Polyethylene (HDPE), Polypropylene (PP) ................................................................................................................................. 11

Table 2: Mechanical properties of Engineering thermoplastics, adapted from [28] Polyurethane (PU), Polyamide 12 (PA 12). .................................................................................................................................................................................... 12

Table 3: Minimum Peak Tensile force for tubular test pieces, adapted from [31]. .................... 13

Table 4: Sample Material Extrusion Dimensions ................................................................................. 22

Table 5: Pulser settings used for Normal US Imaging........................................................................... 26

Table 6: Micro-CT Imaging Settings ..................................................................................................... 28

Table 7: Angled Beam Imaging Settings ............................................................................................. 31

Table 8: Total Normalized Energy Across Groups .............................................................................. 44
ABSTRACT

Endovascular medical devices are composed of multiple thermal plastic components that are typically joined by butt fusion. High quality joining of components is required to provide end patient safety. The current practice for assuring quality of these thermoplastic joints is limited to process validation and destructive testing. Visual inspection is insufficient as subsurface defects can affect joint performance, compromising patient safety. It is hypothesized that subsurface defects which negatively impact weld performance can be detected non-destructively using ultrasound.

We have created a benchtop ultrasonic system for endovascular weld inspection that allows for B-mode imaging and multiangle ultrasound, that is shown to be able to detect faults (porosity and contamination) post welding with a detection limit of between 0.18-0.27mm in polyamide (Pebax® 72D). Ultrasonic inspection is shown to outperform the detection of porosity within a weld, when compared to Micro-CT inspection. Additionally, the bench top ultrasonic system proposed is relatively inexpensive and requires significantly less time to inspect welds (3 minutes) compared to Micro-CT (40 minutes). Ultrasound energy was able to stratify weld quality and is a potential index for in line verification of weld processing. This study demonstrates the feasibility of non-destructive ultrasonic verification to monitor endovascular joining process and allow for 100% verification.
Abrégé

Les dispositifs médicaux endovasculaires sont composés de plusieurs composants thermoplastiques qui sont généralement liés bout à bout par fusion. Des soudures de haute qualité sont nécessaire pour assurer la sécurité des patients. La pratique actuelle pour garantir la qualité de ces joints thermoplastiques se limite à la validation des procédés et à des essais destructifs. L'inspection visuelle est insuffisante car les défauts sous la surface peuvent affecter les performances des joints soudés, mettant en péril la sécurité du patient. L'hypothèse est que les défauts sous la surface, ayant un impact négatif sur les performances de soudage, pourraient être détectés de manière non destructive à l'aide d'ultrasons.

Nous avons créé un système à ultrasons sur table pour l'inspection des soudures d'appareils endovasculaire qui permet une imagerie en mode B et des ultrasons multi angles, qui s'avère capable de détecter les défauts (porosité et contamination) après le soudage, avec une limite de détection comprise entre 0,18 et 0,27 mm dans le polyamide. (Pebax® 72D). Il a été démontré que l'inspection par ultrasons surpasse la détection de la porosité dans une soudure par rapport à l'inspection Micro-CT. De plus, le système à ultrasons de table proposé est relativement peu coûteux, et nécessite beaucoup moins de temps d'inspection des soudures. L'énergie ultrason a permis de stratifier la qualité de la soudure, et constitue une potentielle étape essentielle pour la vérification sur la ligne de production du traitement des soudures. Cette étude démontre la faisabilité d'une vérification par ultrasons non destructive pour surveiller le processus de jonction endovasculaire et permet un contrôle de qualité totale.
<table>
<thead>
<tr>
<th>Term</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>FEP</td>
<td>Fluorinated Ethylene Propylene</td>
</tr>
<tr>
<td>HDPE</td>
<td>High density polyethylene</td>
</tr>
<tr>
<td>ID</td>
<td>Inner Diameter</td>
</tr>
<tr>
<td>IQ</td>
<td>Installation Qualification</td>
</tr>
<tr>
<td>LDPE</td>
<td>Low density polyethylene</td>
</tr>
<tr>
<td>LLDPE</td>
<td>Linear low-density polyethylene</td>
</tr>
<tr>
<td>Micro-CT</td>
<td>X-Ray Microtomography</td>
</tr>
<tr>
<td>NDE</td>
<td>Non-Destructive Evaluation</td>
</tr>
<tr>
<td>OD</td>
<td>Outer Diameter</td>
</tr>
<tr>
<td>OQ</td>
<td>Operational Qualification</td>
</tr>
<tr>
<td>PA 12</td>
<td>Polyamide 12</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>PQ</td>
<td>Performance Qualification</td>
</tr>
<tr>
<td>PU</td>
<td>Polyurethane</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinyl Chloride</td>
</tr>
<tr>
<td>SPL</td>
<td>Spatial Pulse Length</td>
</tr>
<tr>
<td>US</td>
<td>Ultrasound</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate Tensile Strength</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
</tr>
<tr>
<td>--------</td>
<td>---------------------------------</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>Attenuation Coefficient</td>
</tr>
<tr>
<td>$K$</td>
<td>Bulk Modulus</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Density</td>
</tr>
<tr>
<td>$d$</td>
<td>Diameter</td>
</tr>
<tr>
<td>$\Gamma$</td>
<td>Elastic Modulus</td>
</tr>
<tr>
<td>$f$</td>
<td>Frequency</td>
</tr>
<tr>
<td>$Z$</td>
<td>Impedance</td>
</tr>
<tr>
<td>$a$</td>
<td>Intensity Reflection Coefficient</td>
</tr>
<tr>
<td>$c$</td>
<td>Longitudinal Velocity</td>
</tr>
<tr>
<td>$NF_{z}$</td>
<td>Near Field Zone</td>
</tr>
<tr>
<td>$\nu$</td>
<td>Poisson’s Ratio</td>
</tr>
<tr>
<td>$p$</td>
<td>Pressure Plane</td>
</tr>
<tr>
<td>$G$</td>
<td>Shear Modulus</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>Strain</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>Stress</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Wavelength</td>
</tr>
<tr>
<td>$E$</td>
<td>Young’s Modulus</td>
</tr>
</tbody>
</table>
1. INTRODUCTION AND BACKGROUND

1.1 Introduction

Medical devices have a large range of applications. Many devices are highly specialized and are becoming ever more so as technology advances. The design and performance of these devices rely heavily on the materials used and quality of manufacturing. As such, assembling medical grade materials into high quality devices comes at high cost to medical device manufacturers worldwide. Plastics are widely used for many disposable devices as they allow for design flexibility, miniaturization of components, sterilization, ease of processing, and can be thermal insulators, water resistant, chemical resistant, nonallergenic, and more [1].

Due to the highly regulated environment for medical device manufacturing, the output of all manufacturing processes must either be fully verified or undergo process validation. Verification is the preferred monitoring method and typically involves pass/fail criteria or measurements used to differentiate acceptable from unacceptable devices (rejects). However, non-destructive verification is not feasible or possible for all processes and current industry practice relies heavily on process validation.

Commonly validated processes include heat treating, plating, plastic injection molding, extrusion, welding, plastic bonding and soldering [2]. Verification of these processes currently requires destructive testing such as tensile testing, compression testing, kink testing, pressure testing and/or overall performance testing. Such testing is not feasible for 100% of products, as the product would be compromised or destroyed. The validation and re-validation system provides confidence by examining a sampling of the product. This sampling by nature is unable to provide continuous monitoring of all products and processes over time. Process validation is also expensive to implement and can go overlooked. In 2014 alone, there were 122 warnings of FDA regulation violations (FDA 483’s) issued due to lack of or inadequate process validation [3] many of which required corrective actions by the receiving company.
Once a process is determined to required validation (Figure 1), process validation follows a predetermined protocol including installation qualification (IQ), operational qualification (OQ), and performance qualification (PQ) [4]. The objective of IQ is to establish objective evidence that the equipment used in the process is installed as per the manufacturers approved specifications [4]. This is followed by determining process windows usually through small scale testing to determine the acceptable process windows (i.e., parameters) for a specific process along with determination of desired process outputs (e.g., weld performance). For welding, these input parameters would include time, temperature, and pressure while the output typically is ultimate tensile strength. Once the input/output windows are defined, OQ can be conducted. OQ is the act of establishing objective evidence that the equipment used for the process when used within the input parameter windows, produces acceptable results (i.e., worst case testing, high/low parameter testing). Once OQ has been completed and the process has been shown to create acceptable results within the parameter range, the final step is PQ. During PQ, the objective is to demonstrate that the process can consistently produce acceptable results under normal operating conditions. For both OQ and PQ statistically relevant samples sizes are required, and all samples are typically destroyed during the validation process. The example presented in the Global Harmonized Task Force [4] guidance for heat sealing of pouches examined 90 samples during process window definition and 570 samples during OQ alone. Finally, processes require monitoring to ensure a state of control is maintained, and asses whether any changes that could

Figure 1: Process Validation Decision Tree, adapted from [4].
impact the validated state (equipment, location transfer, product design, etc.) may require revalidation [4].

![Figure 2: Balloon catheter example (distal end), weld locations indicated by orange arrows.](image)

Due to the limitations of process inspection and the nature of current industry standards, non-verified devices are being made and distributed. As such, patients worldwide are being exposed to a higher risk of device failure than if every device for every process was indeed checked and verified for quality. This work aims to demonstrate feasibility of a non-destructive evaluation technique that could be used to verify plastic welding process output (see Figure 2) in endovascular devices.

1.2. Objectives of Research

It is hypothesized that polymer welds, within minimally invasive catheters, that contain defects could be identified using non-destructive ultrasound evaluation. Current industry techniques use ultrasonic evaluation to verify the output of pipe welding for large, dense plastic piping. However, the feasibility and application of the technique has not been shown on the very small scale using flexible, biocompatible plastics used in medical devices. The goal of this thesis is to demonstrate that certain weld faults within a polymer welds can be detected using ultrasonic non-destructive evaluation.

The specific objectives of this thesis research are as follows:

1. Create a low-cost bench top ultrasonic setup that can inspect catheter welds.
2. Create catheter weld conditions with representative common industry fault conditions that decrease weld performance.

3. Perform high resolution x-ray microtomography of endovascular catheter welds to detect the presence of faults within the welds and their impact on weld performance.

4. Perform ultrasonic evaluation of endovascular catheter welds to detect the presence of faults within the welds and their impact on weld performance.

5. Benchmark the fault detection capability of the ultrasonic evaluation with high resolution x-ray microtomography.
2. CURRENT LITERATURE

Current verification of processes relies on non-destructive testing such as visual inspection, dimension measurements, and resistance testing. The following provides an overview of the current methodologies used for non-destructive evaluation (NDE) and their suitability to detect faults in plastic welds of the most used biocompatible plastics, specifically in minimally invasive medical devices such as catheters.

2.1. Non-Destructive Evaluation

A variety of NDE methods were identified and the compatibility to catheter bond inspection were examined. The list of techniques investigated includes:

- Acoustic Emission
- Eddy Current Techniques
- Shearography
- Infrared Thermography
- Electromagnetic Waves
- X-Ray Microtomography
- Ultrasound

Acoustic emission is a transient elastic wave generated by rapid energy release from a local source within a material under stress [5]. The emission is generated by fracture, crack initiation and growth, structural separation, and movement between material phases. By applying stress to the material in a ramp fashion, the number of acoustic emissions accelerate steadily for non-structurally damaged material. By applying sub maximum tensile strength, the overall integrity of the material is not substantially compromised. However, in faulty, inhomogeneous materials, the number and intensity of the acoustic emission events is notably different [5]. Since this NDE method requires that the part be microscopically damaged to be evaluated, it is not suitable for the examination of medical device welds where fatiguing a component may subject its users to undue risk.

Eddy Current Techniques function by measuring variation to electric and dielectric properties of the material [6]. They are used to characterize the surface of a material through generation of eddy currents when a magnetic field, formed using alternating current and an induction coil, is
placed near the surface in question. The eddy currents generated by the material create a secondary magnetic field which opposes the original field. This reduction is based on the conductivity of the material and causes an impedance change which is measured by a pick up coil [6]. Though this technique could be applicable to some medical devices, most catheters are made of non-conductive plastics. As such, eddy current techniques are not well suited for the evaluation of medical device polymers.

Shearography is an optical technique to visualize variations of materials response in a loaded and unloaded state [7, 8]. By observing the speckle pattern formed by coherent laser light of a surface at rest and then comparing it to a surface under load, deformities can be identified. This identification is on the basis that areas with defects will perform differently under load than non-defective areas. This loading can be done thermally, under vacuum, through vibration excitation or through mechanical excitation [7, 8]. Evaluation is limited to materials under strain and their response to varying strain. As this technique requires the samples to be loaded externally, it puts the component at risk of unknown effects of fatigue.

Thermography is a technique that measures the surface temperature of a material as heat flows through it. It detects flaws by finding hot or cold locations that are indicative of an area that is unlike the surrounding material [9, 10]. These areas can be caused by flaws in the material which lead to variations in heating or cooling rate. Thermography can be applied passively by examining the substrate during normal use conditions or actively by applying heat to the area of interest. The passive evaluation compares the object’s heat distribution against a thermal map of a fault free system. The size of defects that can be detected is given by $2d \geq z$, where $z$ is the defect depth and $d$ is the diameter of the defect [9, 10]. This means a defect at 0.5mm depth within a device would have a minimum diameter of 1mm to be detected. This scaling makes thermography poorly suited for evaluation of small medical devices.

NDE using electromagnetic waves (common referred to as microwave imaging) uses frequencies in the range of 0.3GHz to 30GHz. These waves can penetrate non-conductive materials such as wood, ceramic, concrete, and polymeric materials. As microwaves traverse through a material, the wave is affected by its relative permittivity (dielectric constant). Therefore, microwave testing
detects and displays local variations of the dielectric constant in non-conductive materials [11]. The spatial resolution of these systems follows the principles of wave propagation and is therefore limited to approximate half the wavelength in the far-field. At the highest frequencies (300GHz) the wavelength is approximately 1mm providing a spatial resolution of 0.5mm [12, 13]. The near-field resolution is proportional to the sensor sensing area. Channel defects as small as 0.01mm were detected by Morita et al. [14]. This method could be a viable means of detecting faults in non-conductive medical devices, however it has been limited to laboratory applications so far due to the length of scanning time required [15].

X-Ray Microtomography (Micro-CT) is a 3D imaging technique that scans objects in 2D planar images and uses offline reconstruction to create 3D imaging. As X-ray photons pass through an object, differences in absorption cause variations in intensity. The object is then rotated and scanned again at 1-5° increments until a 180° image can be created. This technology allows for visualization of defects within an object and can provide a resolution down to 100 nanometers [16]. The limitation of this NDE method are the high capital costs required for the system, limited sample space/field of view and the required inspection time. With its high accuracy and ability to measure 3D geometries, Micro-CT is widely used for patient specific analysis and implant creation [17, 18], but is impractical for in line manufacturing verification.

Ultrasonic NDE testing is currently used on a wide variety of applications. It is present in the aviation and automotive industries, ensuring the quality of metallic welds. It is also used to evaluate polyethylene (PE) and high-density polyethylene (HDPE) piping used in Nuclear Power Plants [19-21]. It has been shown to be feasible as an inspection technique for HDPE and PVC water pipelines for groves down to 1mm in width [22]. In this thesis, the suitability for ultrasonic testing of medical device welds will be evaluated. Ultrasound (US) operates using acoustic energy traveling through a medium in order to resolve information about the medium traversed. US waves are defined by the incident pressure, the US wave velocity, the frequency, and the wavelength among other properties. The propagation of US waves is heavily dependent on the propagation medium and is scattered when interfaces between materials are reached. This interface detection makes it suitable to identify defects in polymers [11, 23], as malformed parts alter the US wave differently than correctly formed parts. The resolution of ultrasound is highly
dependent on the transducer(s) used but the successful detection of faults in packaging tray seals ≥0.02 mm using a 20MHz transducer has been reported [24]. The limitations of ultrasonic NDE are that a coupling agent is required, and that a high degree of skill is required to interpret results [15]. This method could be a viable means of detecting faults in polymers used in medical devices and is the method selected for this thesis.

2.2 Medical Device Polymers

Materials used in medical devices are required to not only perform their specific function, but also to meet regulatory standards with regards to multiple other aspects. These aspects include shelf life and stability, sterilization resistance, chemical and lipid resistance, biocompatibility, hemocompatibility, and non-toxicity. Materials used consist of metals, alloys, ceramics, glass, and plastics. These requirements are enforced by regulatory bodies around the world and as such, many devices used for invasive surgeries are fabricated largely out of plastics. Plastics are high-molecular weight polymers that can be formed into a range of different products such as coatings, films, and solid components. The use of plastics allows for many advantages from a design standpoint. They can be created on very small scales with tight tolerances and can be lightweight, electrical/thermal insulators, and can be bonded to other materials. Additionally, many plastics are inherently biocompatible or can be made as such [1].

In addition to the regulatory requirements, each device is designed to perform specific tasks and as such have individual device requirements. These needs shape the design of the device and the selection of materials used. For catheter applications generally, the following requirements apply; flexibility, durability, kink resistance, extrusion processability, columnar strength, tensile strength, and biocompatibility. To meet these requirements, there are many different types of plastics that can be used.

Plastics can be classified in three groups, thermoplastics, thermosets, and elastomers (Figure 3). Thermosets are typically formed in an irreversible process and create rigid structures. Elastomers are loosely cross-linked polymers that are rubbery and used when flexibility and elasticity is needed. For example, gaskets, seals, and rubber septums are typically fabricated from elastomers. This review will be limited to thermoplastics that are commonly bonded together to
form a medical device. The most common plastics used for this purpose, commodity plastics and engineering thermoplastics, will be examined. Commodity plastics account for roughly 80% of plastics used for medical devices [1]. These plastics included Polyvinyl Chloride (PVC), Linear low-density polyethylene (LLDPE), low density polyethylene (LDPE), HDPE and polypropylene (PP). PVC is the most common plastic used as it is found in 25% of all medical devices. Common uses include: blood bags, tubing, gloves, dialysis equipment, mouthpieces, masks, catheters, injection moulded parts, and device packaging [25]. Flexible PVC is often used in catheters. In all plastic formulations, the main polymer is only one component and can comprise of less than 30% of overall composition. The other additives are plasticizers, heat stabilizers, processing, aids, colours, or other polymers creating blends. LDPE is commonly used for tubing, catheters, packaging, IV fluid bottles, caps for luers and bottles, and under pads for hospital beds. It is easier to weld than HDPE. HDPE is commonly used for filters, clamps, and heart valves. PP is used for packaging, pouches, drapes, gowns, sutures, and syringes. The thermoplastics that are conducive to welding and represent a large percentage of the medical device market.

Engineering thermoplastics are named as such as they have properties that yield improved performance when compared to commodity plastics. Engineering thermoplastics that offer properties suitable for the use in catheters include Polyester Polyurethane (Polyester PU), Silicone PU, Polyamide 12 (PA 12), Nylon (4, 6). Table 1 and Table 2 display the material properties. PU’s (Polyester PU and Silicone PU) and Polyamides (Nylon, PA 12, Pebax®) are used for catheter and catheter component construction such as occlusion balloons. Performance classification is visualized in Figure 4.
Figure 3: Polymer Classifications, adapted from [1]

Figure 4: Thermoplastic classification, adapted from [1]
Table 1: Mechanical properties of commodity plastics, adapted from [25-27]. Polyvinyl Chloride (PVC), Linear Low-Density Polyethylene (LLDPE), Low Density Polyethylene (LDPE), High Density Polyethylene (HDPE), Polypropylene (PP)

<table>
<thead>
<tr>
<th></th>
<th>PVC-unplasticized</th>
<th>PVC-Plasticized (40% DEHP)</th>
<th>LLDPE</th>
<th>LDPE</th>
<th>HDPE</th>
<th>Isotactic-PP</th>
</tr>
</thead>
<tbody>
<tr>
<td>% of Devices</td>
<td>~75</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>1.38-1.4</td>
<td>1.2-1.3</td>
<td>0.92</td>
<td>0.91-0.93</td>
<td>0.94-0.97</td>
<td>0.905</td>
</tr>
<tr>
<td>Melting point (°C)</td>
<td>170-180</td>
<td>170-180</td>
<td>102-125</td>
<td>100-110</td>
<td>130-135</td>
<td>163</td>
</tr>
<tr>
<td>Glass Transition Temp (°C)</td>
<td>80</td>
<td>-40-20</td>
<td>-110</td>
<td>-110</td>
<td>-90</td>
<td>-10</td>
</tr>
<tr>
<td>Heat Distortion Temp @ 1.8MPa (°C)</td>
<td>60-75</td>
<td>20</td>
<td>35</td>
<td>30-40</td>
<td>38-50</td>
<td>55</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td>45-55</td>
<td>10-20</td>
<td>11</td>
<td>8-15</td>
<td>18-30</td>
<td>30-35</td>
</tr>
<tr>
<td>Elongation at Break (%)</td>
<td>20-100</td>
<td>100-500</td>
<td>300-900</td>
<td>90-800</td>
<td>20-500</td>
<td>100-300</td>
</tr>
<tr>
<td>Flexural Modulus (GPa)</td>
<td>2-5</td>
<td>0.01-0.03</td>
<td>0.15</td>
<td>0.25</td>
<td>0.8-1.25</td>
<td>1.5-2.0</td>
</tr>
<tr>
<td>Impact Strength (notched, J/m)</td>
<td>20-100</td>
<td>90-110</td>
<td>50-1000</td>
<td>No break</td>
<td>50-100</td>
<td>50-120</td>
</tr>
<tr>
<td>Crystallinity (%)</td>
<td>-</td>
<td>-</td>
<td>30-40%</td>
<td>40-50</td>
<td>60-80</td>
<td>40-60</td>
</tr>
<tr>
<td>Bulk Modulus (GPa)</td>
<td>5.47</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Youngs Modulus (N/mm²)</td>
<td>4440</td>
<td>-</td>
<td>110-450</td>
<td>1035</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Poisson Ratio</td>
<td>0.37</td>
<td>-</td>
<td>-</td>
<td>0.4-0.45</td>
<td>0.43</td>
<td></td>
</tr>
<tr>
<td>Attenuation Coefficient (dB/mm)</td>
<td>1.24 (2MHz)</td>
<td>-</td>
<td>-</td>
<td>0.9 (3MHz)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Longitudinal Ultrasound Velocity (m/s)</td>
<td>2353</td>
<td>-</td>
<td>-</td>
<td>2265 @ 3MHz</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>
Table 2 Mechanical properties of Engineering thermoplastics, adapted from [28] Polyurethane (PU), Polyamide 12 (PA 12).

<table>
<thead>
<tr>
<th></th>
<th>Polyester PU</th>
<th>Silicone PU</th>
<th>PA 12</th>
<th>Nylon (4,6)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Density (g/cc)</strong></td>
<td>1.07-1.25</td>
<td>1.05-1.2</td>
<td>1.01</td>
<td>1.18</td>
</tr>
<tr>
<td><strong>Glass Transition Temp (°C)</strong></td>
<td>-</td>
<td>-</td>
<td>41</td>
<td>78</td>
</tr>
<tr>
<td><strong>Softening Point (°C)</strong></td>
<td>50-80</td>
<td>65-85</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>Melting Point (°C)</strong></td>
<td>-</td>
<td>-</td>
<td>177</td>
<td>295</td>
</tr>
<tr>
<td><strong>Heat Distortion Temp @ 1.8 MPa (°C)</strong></td>
<td>-</td>
<td>-</td>
<td>42</td>
<td>160</td>
</tr>
<tr>
<td><strong>Tensile Strength @ Break (MPa)</strong></td>
<td>20-55</td>
<td>25-55</td>
<td>45-52</td>
<td>7</td>
</tr>
<tr>
<td><strong>Tensile Elongation @ Break (%)</strong></td>
<td>50-950</td>
<td>350-900</td>
<td>275-325</td>
<td>25</td>
</tr>
<tr>
<td><strong>Flexural Modulus (GPa)</strong></td>
<td>0.025-0.5</td>
<td>0.03-0.04</td>
<td>0.8-1.2</td>
<td>3.7</td>
</tr>
<tr>
<td><strong>Processing Temperature (°C)</strong></td>
<td>50-235</td>
<td>170-210</td>
<td>200-220</td>
<td>300-320</td>
</tr>
</tbody>
</table>

2.3. Types of Faults
Polymer welding when completed correctly, creates a smooth transition between components and mechanically joins them together. Weld integrity depends on three main parameters: temperature, exposure time, and pressure. The material needs to be exposed to sufficient weld temperatures for a long enough period so that it reaches the desired temperature (i.e. the melting point). If the joint is under too high or too low pressure, mixing of the polymers is not possible. Interfaces must also be free of contamination such as dust or oil as it interferes with the polymer mixing process. If the welds are not correctly created many different types of fault may occur that can impact weld strength. Fault types include cracks, cavities, solid inclusions, lack of fusion (cold welding or contamination), form failure and thermal damage. Figure 5 displays some of these fault types. To reliably inspect plastic welds, the inspection technique should be able to detect all fault types that impact joint performance. Identification of the specific fault is not required but a correct weld should be able to be distinguished from a faulty weld.

One of the largest challenges for many NDE techniques is the identification of cold welding joints, where the joint was not sufficiently heated to allow for complete polymer mixing. The Eigen line indicates the transition region between the weld affected area and to the non-weld affected part. The distance between the Eigen line and the weld line shows a certain relationship with the welding strength in joints. Crystallinity and elastic modulus would be higher in the base material than the weld due to the forming conditions [29].
The scale of medical devices could also pose a challenge. Looking at intravascular catheters, the maximum outer diameter is commonly measured in French (Fr, 1 Fr = 1/3 mm). The sizes vary depending on application and the scale ranges between 3-34 Fr (1 – 11.3mm). Representative samples will have to be investigated to learn the approximate size of defect that significantly affects the performance of the weld. International standards dictate the minimum peak tensile force required for each catheter test piece with respect to their minimum outer diameter (OD), shown in Table 3 [31]. However, each piece may have elevated tensile strength requirements based on their specific function or use conditions and any decrease in weld performance could compromise the safety and efficacy of the device. The impact of each type of fault should be investigated. Many studies have been conducted examining the maximum tensile strength [24, 32-34] of bonds and optimizing the welding parameters but to our knowledge, little has been done to examine the effect of specific faults on predicting bond performance for endovascular devices.

**Table 3:** Minimum Peak Tensile force for tubular test pieces, adapted from [31].

<table>
<thead>
<tr>
<th>Smallest OD of Test Piece</th>
<th>Minimum Peak Tensile Force (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>≥ 0.55 &lt; 0.75 mm</td>
<td>≥ 1.7 &lt; 2.3 Fr</td>
</tr>
<tr>
<td>≥ 0.75 &lt; 1.15 mm</td>
<td>≥ 2.3 &lt; 3.5 Fr</td>
</tr>
<tr>
<td>≥ 1.15 &lt; 1.85 mm</td>
<td>≥ 3.5 &lt; 5.6 Fr</td>
</tr>
<tr>
<td>≥ 1.85 mm</td>
<td>≥ 5.6 Fr</td>
</tr>
</tbody>
</table>
2.4. Ultrasonic Testing

2.4.1. Operation Principles

US testing operates using acoustic energy traveling through a medium in order to resolve information about the medium traversed. US waves are defined by the incident pressure, the US wave velocity, the frequency, and the wavelength among other properties. The propagation of US wave depends on the propagation medium. The properties of the medium of interest are its density, temperature, impedance, and attenuation. US waves can occur as longitudinal waves (compressional) or as shear waves. As the US energy is traversing the medium, regions of compression and rarefaction occur because of sinusoidal wave propagation. The definition of the wavelength is described as:

\[ \lambda = \frac{c}{f} \]  
Equation 1

Where \( \lambda \) is the wavelength expressed in meters, \( c \) is the velocity of wave energy through the medium in meters per second and \( f \) is the frequency expressed in Hertz. The speed of sound through a medium is dependent on Young’s modulus (\( E \)) and the density of the material, \( \rho \), as given in Equation 2. As temperature is increased, mechanical relaxation occurs and the density decreases, which leads to an increase in sound velocity.

\[ c = \sqrt{\frac{E}{\rho}} \]  
Equation 2

As the wave travels through the medium, it experiences stress and strain whose relationship can be described using Hooke’s Law [35].

\[ \sigma = \Gamma \varepsilon \]  
Equation 3

Where stress (\( \sigma \)) is the force per unit area, strain (\( \varepsilon \)) is the dimensionless expansion per unit length, and the elastic modulus (\( \Gamma \)) relating the stress to the strain. There are three different
types of elastic moduli depending on the type of deformation experienced for linear elastic materials. There is Young’s modulus, the shear modulus (G) and the bulk modulus (K). Young’s modulus, Equation 4 [35], is defined when a normal (perpendicular to the surface) stress ($\sigma_N$) creates a normal strain ($\varepsilon_N$).

$$\sigma_N = E\varepsilon_N$$  \hspace{2cm} \text{Equation 4}

The shear modulus describes when the shear strain is tangential to the surface and the bulk modulus describes the effects of a compression or inward pressure on the object. The elastic modulus is also related to the speed of sound waves in a medium, Equation 5 [35].

$$c = \sqrt{\frac{\Gamma}{\rho}}$$  \hspace{2cm} \text{Equation 5}

For longitudinal waves, particle motion is parallel to the direction of the wave. The wave speed in a fluid is defined using the bulk modulus [35].

$$c = \sqrt{\frac{K}{\rho}}$$  \hspace{2cm} \text{Equation 6}

Shear waves have a particle motion which is perpendicular to the direction of wave and are defined by the shear modulus[35].

$$c_s = \sqrt{\frac{G}{\rho}}$$  \hspace{2cm} \text{Equation 7}

These moduli are not independent, as they are directly related to the property of the material to regain its original shape and orientation after deformation. Therefore, the relationship between
the Youngs modulus, Shear modulus, and bulk modulus can be described by Poisson’s ratio \( (v) \) [36] if the material is isotropic and linear.

\[
E = 2(v + 1)G = 3K(1 - 2v)
\]

Equation 8

As US energy travels through a material, it undergoes refraction and reflection at each interface. Welds incorporate a mixing of materials, if not well welded, interfaces between these materials will provide an interface for US energy to be reflected. Impedance is defined as the resistance of material to deformations. The relative amplitudes of transmission and reflection at a boundary can be described using the impedance of the materials. Reflection refers to energy that is not transmitted through the boundary, while refraction is the energy that is transmitted. The angle of transmission can be calculated using Snell’s Law. The pressure and medium velocity must be continuous and energy is conserved across the boundary [37].

**Figure 6**: Snell’s Law representation adapted from [37]. Material impedance difference \((Z_a \text{ and } Z_b)\), incident wave angle \((\theta_i)\), reflected wave angle \((\theta_r)\), transmitted wave angle \((\theta_t)\)

US images are formed using the reflected echoes in addition to the diffusely scattered echoes due to small inhomogeneities found within welds.
As US waves travel through a medium, the medium resists deformation. This resistance is the impedance of the material. The acoustic impedance, \( Z \) (kg/m\(^2\)s), is equal to the product of the density, \( \rho \), and velocity of sound in the material.

\[
Z = \rho c
\]  
Equation 9

The intensity reflection coefficient \( (a) \), the ratio of the reflected wave relative to the incident wave is shown in Equation 10.

\[
a = \frac{(Z_b - Z_a)^2}{(Z_a - Z_b)^2}
\]  
Equation 10

Sound velocity increases with density while the transmission coefficient decreases [38]. The transmission coefficient describes the amplitude of the refracted acoustic wave relative to the incident wave.
2.4.2. Transducers

US pulses are created using transducers. By applying an electrical charge to a piezoelectric element, it mechanically deforms. This produces the acoustic energy that travels through the material in question. The inverse process, mechanical deformation generates a net electrical charge, allowing for measurement of ultrasonic pulses. The simplest of ultrasonic transducers is the single element piston transducer, shown in Figure 7.

Single transducers provide a very limited field of view providing a single A-Line and would require scanning to examine a weld thoroughly. The resolution of that line is discussed in the following section. Linear array and phased arrays allow for digital beam steering that enables more control over the imaged area and can provide better lateral resolution due to apodization of aperture size to maintain beam width throughout the depth of view (Figure 8).
2.4.3. Resolution

Ultrasound resolution is dependent on the frequency, amplitude and transducer used. Spatial resolution for an ultrasonic test is defined as the smallest distance between two points that can be distinguished. Axial (or longitudinal) resolution is the ability to distinguish points parallel to the US beam while lateral resolution is for points perpendicular to the beam. For a single transducer the axial resolution is the half the spatial pulse length (SPL) [40]. The SPL is the amount of space the driving wave occupies, measured from the beginning of the pulse to the end of the pulse. The SPL is dependent on the frequency of the driving wave, the pulse duration (number of cycles) and the speed of sound in the material. Axial resolution is high when SPL is short. Therefore, axial resolution can be improved by increasing excitation frequency and in materials of higher density.
Lateral resolution varies with respect to depth due to changes in the beam profile. It is most precise where the beam is focused. The beam profile is separated into two zones, the near zone (Fresnel Zone) and the far zone (Fraunhofer’s Zone). The width of each zone is dependent on the size of the transducer face. The beam width in the near field is approximately the same size as the transducer face. The beam converges to a focal point that is approximately half the diameter of the transducer and then begins to diverge in the far zone. Lateral resolution is high when the near zone ($N_F z$) length is long and can be approximated using Equation 11 [40].

$$N_F z = \frac{d^2}{4\lambda}$$  \hspace{1cm} \text{Equation 11}

Where $d$ is the diameter of the transducer and $\lambda$ is the wavelength at the excitation frequency. Transducer arrays can decrease the size of the active transducers, shortening the near field zone and changing the focal point of the transducer. As stated above, catheters range between 1-11.3mm in diameter and the size of defects that impact performance for the specific application would require investigation. As the samples to be measured will be stationary, temporal resolution and Doppler effects will not be examined.

As the US wave travels through the medium, it loses energy through attenuation. Attenuation is frequency dependent and caused by multiple sources such as reflection, refraction, scattering, and absorption. Reflection and refraction can redirect the US wave away from the receiver. Scattering is the effect of mechanical energy being redirected by individual objects and their relative size compared with the incident wave. Absorption is the transfer of mechanical energy into thermal energy. Attenuation is often described through an attenuation coefficient ($\alpha$, dB/cm) [37].

$$\alpha = \frac{1}{z} \ln \frac{p(z = 0)}{p(z)}$$  \hspace{1cm} \text{Equation 12}
Where $p$ is the pressure plane of a monochromatic wave traveling in the $z$ direction, $p(z=0)$ is the pressure at $z=0$, and $z$ is the distance from the source [37].

US NDE is currently in use for industrial inspections to detect weld faults. As good welds are continuous and homogeneous, US is well suited to react to any impedance changes or discontinuities (faults) hidden within a weld. It has been shown to detect tray seal faults $\geq 0.02$ mm [24] and we think that US is well suited for the creation of a relatively inexpensive system that is highly sensitive to discontinuities within endovascular catheter welds.
3. METHODS

To image catheter welds, an ultrasound imaging setup was created, and the accuracy of the resultant images were demonstrated. In order to check the overall functionality, samples were both imaged using US and Micro-CT. Micro-CT was chosen as the gold standard as it is known for high resolution imaging. Using the calibrated results from the Micro-CT, the functionality of the US setup was shown in the normal imaging case. Weld inspection was then examined using angled beam imaging.

3.1. Sample Creation

The most common types of medical grade thermoplastics used in catheters are polyurethanes (e.g. Pellethane®, Tecothane™) and polyamides (e.g., nylon, Pebax®, Vestamid®, Grilamid®). Both polyurethanes and polyamines are engineering thermoplastics. They come in a range of durometers depending on the intended application. Often, similar materials are welded together (i.e., a compliant balloon mounted on a stiffer shaft). As these plastics are acoustically similar to water, the transmission coefficients should be quite high. As a representative base case, a single lumen Pebax® 72D tube was butt welded together to form a continuous piece (Figure 10). Samples were transparent for ease of fault creation, however both Micro-CT and US can be performed on coloured or opaque tubing commonly used for commercial endovascular devices.

<table>
<thead>
<tr>
<th>Name</th>
<th>OD (mm)</th>
<th>ID (mm)</th>
<th>Wall Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pebax® 72D</td>
<td>2.4</td>
<td>1.5</td>
<td>0.45</td>
</tr>
</tbody>
</table>

Samples were welded together using hot air and rotated manually with an inner mandrel (1.47 mm OD) and outer jacket (FEP fusing sleeve – 1.905 mm minimum recovered ID) for support.
Three groups of samples were created: control, porous, and contaminated to examine two common weld faults. All three groups were created using a Weller hot air rework station (WRS1002) at 215°C, 22% Air Flow and welded between 30-45s. The pressure is provided by the outer jacket, which shrinks down when heated and maintains the geometry of the weld. The porous samples were wetted with alcohol underneath the outer jacket to create voids within the welded area. The contaminated samples had talcum powder added to the interface of the weld as a form of contamination. Once cooled to near ambient (3 minutes), the mandrel and outer jacket were removed.

Figure 11: (A) Hot air welding of samples using inner support mandrel and outer FEP heat shrink sleeve. (B) Representation of sample creation.
3.2. Ultrasound Setup Design

An ultrasound probe was driven using a pulser receiver (JSR DPR300) and its output was read by an oscilloscope (Siglent SDS 1102X, 1GSa/s) and saved as an averaged (32 traces) radiofrequency response by the desktop computer. The signal underwent offline processing using Matlab™. Movement of the transducer was handled by a motor assembly consisting of two perpendicular translation stages (Velmx – BiSlide Linear Stage) allowing for horizontal translation along the weld, and vertical displacement for focusing. Each stage includes a stepper motor (Slo-Slyn Motor, M062-LS-554) which are independently controlled by a single motor controller (Velmex, VXM). These stages are capable of displacing at a resolution of 0.00635 mm and are operated externally via serial command. Sample rotation was performed with a stepper motor (28BYJ-48) capable of 0.0879° steps controlled via Arduino. Fixturing was also created to accommodate test samples of various sizing, stabilize test piece positioning, and allow for varied probe angles. The test piece and probe were submerged in water (tap water, room temperature) to act as a coupling medium. An image and diagram are presented in Figure 12 and Figure 13, respectively. A 15MHz spherical point focus transducer (Olympus V3119) was used with a beam diameter of 0.2mm, axial resolution of 0.13mm (in Pebax® 70D).
Figure 13: Experimental Setup Diagram

Thirty-two signal traces were averaged by the oscilloscope to improve the signal-noise ratio. The average signals were then stored on the computer for offline processing. The overall cost of the equipment used in the setup was less than $7000 USD.
3.3. **Setup Verification – Normal Incidence US Imaging**

To verify the US setup, wall thickness measurements were acquired for the control group. The probe was positioned perpendicular to the test piece and advanced in 0.1mm steps along the sample to image the length of the weld. This imaging mode is limited to recording a single plane of the test piece and thus would provide limited information regarding the full weld integrity, but the aim was to verify the setup, including the offline image processing. The US probe was driven using the Pulser Receiver with the settings given in Table 5.

**Table 5: Pulser settings used for Normal US Imaging**

<table>
<thead>
<tr>
<th>Pulser Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulse Repetition Frequency</td>
<td>100Hz</td>
</tr>
<tr>
<td>Voltage</td>
<td>100 V</td>
</tr>
<tr>
<td>Damping</td>
<td>44 Ohms</td>
</tr>
<tr>
<td>Ext. Trigger Rin</td>
<td>50 Ohms</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Receiver Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bandwidth</td>
<td>50MHz</td>
</tr>
<tr>
<td>Gain</td>
<td>50dB</td>
</tr>
<tr>
<td>Low Pass Filter</td>
<td>50MHz</td>
</tr>
<tr>
<td>High Pass Filter</td>
<td>2.5 MHz</td>
</tr>
</tbody>
</table>

The total distance traveled by the probe was 9mm. At each step, the oscilloscope recorded 7000 points, averaged 32 times at a sampling rate of 1G samples/second. The oscilloscope gating was set at the transducer focal point with a delay of 26.4μs (center). Signals were then processed in Matlab™ to measure the top wall thickness of the weld. Processing steps include, digitally filtering the samples using a 4th order Butterworth filter (3-30MHz), converting to greyscale, resizing the image for visualization, thresholding to isolate wall reflections, and then calculating the distance in pixels between the inner and outer top wall reflection, all done using the image processing tools in Matlab™. This time of flight (TOF) measuring approach converted the pixel count to mm.
using the speed of sound in water, 1481m/s at 20°C. The calculated wall thickness was then compared to the wall thickness measurements using the Micro-CT.

Figure 14: (A) Raw US Signal, top and bottom wall shown (B) Signal processed for top wall thickness measurements.
3.4. **Micro-CT Weld Inspection**

Micro-CT imaging was performed on the three groups of samples using a Bruker Skyscan 1172. The settings used can be found in Table 6. Images were reconstructed using SkyScan NRecon and 3D analysis was performed using SkyScan CTAN. Single 2D scans for each group are shown in Figure 15, Figure 16, and Figure 17.

**Table 6: Micro-CT Imaging Settings**

<table>
<thead>
<tr>
<th>Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source Voltage</td>
<td>37 kV</td>
</tr>
<tr>
<td>Source Current</td>
<td>234uA</td>
</tr>
<tr>
<td>Image Pixel Size</td>
<td>3.99µm</td>
</tr>
<tr>
<td>Rotation Step</td>
<td>1°</td>
</tr>
<tr>
<td>Total Rotation</td>
<td>180°</td>
</tr>
</tbody>
</table>

*Figure 15: 2D Scan Example of Control Group.*
For the control group, wall thickness analysis was performed to compare against the top wall thickness observed in the US setup. This was done using the reconstructed cross-sectional images. The images were then thresholded in SkyScan CTAN and processed in Matlab™. The OD and ID of the tube was detected using the image processing toolbox in Matlab™. As the ID of the tube is not perfectly centered, each image produces a wall thickness range for each position along the sample. The distance between images and the pixel resolution was both 3.99µm. See Appendix: Software for functions used.
Volumetric analysis using density thresholding of each sample was conducted to determine the relative volume of voids and contamination present in each of the samples. Analysis was performed on reconstructed planes.
3.5. **Weld Inspection - Angled Beam Imaging**

As the normal US imaging provided only limited information on defects, restricted to a single plane of the weld, angled inspection was performed. The probe was angled 45° relative to the test piece, focused on the weld interface, and held stationary. This produces both longitudinal and shear waves in the test piece. The test piece was then rotated in 15° increments 24 times. The rotation step size for the test pieces equates to an inner and outer arc length of 0.098 mm and 0.157 mm, respectively. In this orientation, the reflected energy received at the probe is much smaller. The pulser settings are shown in Table 7.

**Table 7: Angled Beam Imaging Settings**

<table>
<thead>
<tr>
<th>Pulser Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulse Repetition Frequency</td>
<td>100 Hz</td>
</tr>
<tr>
<td>Voltage</td>
<td>225 V</td>
</tr>
<tr>
<td>Damping</td>
<td>44 Ohms</td>
</tr>
<tr>
<td>Ext. Trigger Rin</td>
<td>50 Ohms</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Receiver Setting</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bandwidth</td>
<td>50 MHz</td>
</tr>
<tr>
<td>Gain</td>
<td>50 dB</td>
</tr>
<tr>
<td>Low Pass Filter</td>
<td>50 MHz</td>
</tr>
<tr>
<td>High Pass Filter</td>
<td>2.5 MHz</td>
</tr>
</tbody>
</table>

In this orientation, the resultant image produced is not meaningful but rather the amplitude and total energy of the reflected signal are. In welds that are continuous, without inclusions, the reflected signal is expected to be very low, however, when an inclusion or incomplete weld is present the amplitude of the signal would be much greater.

Reflected signal energy was recorded based on the voltage created by the US transducer and read via the oscilloscope. Signal energy was estimated through summation of the squared signal magnitude [41]. As the resistance is fixed but unknown, the energy is presented as normalized against the energy received from a half sample where no weld was present. The end of the tube
allowed for a strong reflection and was used to focus and position the transducer (Appendix: Additional Figures - Figure 38). Bandpass filtering of received signals was performed with a pass range of 2.5-30 MHz using a 4th order Butterworth filter. Signals were processed offline using built in Matlab™ signal processing toolbox and custom functions (see Appendix: Software).
3.6 **Mechanical Testing**

To evaluate the impact of voids and contamination on ultimate tensile strength, and elongation at break samples were pulled until failure using a Tensile Tester (Ametek LRX 5K with a LRX Loadcell 2500 [±1% of reading]). Samples were gripped on either side of the weld such that the gauge length was approximately 25mm for all samples and pulled at speed of 500mm/min. The ultimate tensile strength, the elongation at break, and the failure mode was recorded for each of the samples.

Samples were pulled until break to examine what impact the detected faults had on the performance of the welds. The setup is shown in Figure 19.

![Figure 19: Tensile Testing Failure Mode Examples of A) Control B) Porous C) Contaminated](image-url)
3.7. **Statistics**

Box and whisker graphs depict the mean, minimum, maximum, 25\(^{th}\), and 75\(^{th}\) percentile. Data is assumed to be normally distributed with low sample size. Difference in groups was assessed using One-way ANOVA with a Bonferroni post test. Differences were considered significant at \(P<0.05\). Correlation was considered non-zero at \(P<0.05\). Linear regression lines are presented with 95\% confidence bands (black dotted lines). Cut off values are calculated using the control group mean result plus 3 standard deviations for an expected population coverage of approximately 99.9\%. Wherein 99.9\% of controls (acceptable welds) would exhibit a total inclusion percentage and subsequent energy response below the cut off value. The separation value was then calculated as a factor of the lowest response (inclusion percentage or energy) seen from a faulty sample divided by the cut off value. A larger separation value indicates that there is a larger sensitivity and faults are more readily distinguishable from the control group.
4. RESULTS

4.1 Setup Verification

To verify the setup the wall thickness measured using the normal US TOF imaging was compared to the wall thickness measured using the Micro-CT scanner. To measure the single plane wall thickness of the normal US, the signals were filtered, converted to grey scale images, thresholded and the outer and inner walls were detected. This approach examined one section perpendicular to the transducer. Wall thickness was calculated using the speed of sound in water (1481 m/s at 20°C) and the sampling rate (1G samples/s). The transducer was moved a total of 9mm at 0.1mm increments. For the Micro-CT, the cross-sectional images were used to measure the wall thickness. These images were density thresholded and the ID and OD of the tube detected. The variation in ID centering and outer weld geometry resulted in a range in wall thickness at each measured location. The average of the wall thickness at each position was used for comparison with the measured US wall thickness. Image registration between the field of view in the Micro-CT and the US at the start position and radial location was not controlled across tests so the wall thickness line measured using the US was compared against the average wall thickness across the sample as measured by the Micro-CT. To align the measurements, the wall thickness results were first cross correlated and then the specific sample delay was applied (Figure 20). The aligned results were then compared by examining the root mean square error (RMSE) to provide an estimate of how comparable the US was to the Micro-CT. This comparison produced a RMSE of 0.035mm (n=4) or 7.8% of the nominal tube wall thickness. This resolution is within ½ of the wavelength used and smaller detail would not be expected to be resolved with the 15MHz transducer used.
4.2 Weld Performance

Three sample groups were created (controls, porous, and contaminated) as outlined in 3.1 Sample Creation. During sample creation, 6 of both the control and contaminated samples were made. A total of 7 porous samples were created, however one sample had visually apparent surface cavities and defects which would be detected using standard industry post process visual inspection and was thus not used for subsequent analysis. Additionally, one control sample was mishandled during mechanical testing, and could not be tested. Therefore, a sample size of n = 6 for each defect sample group (porous and contaminated) group was tested and a sample size of n = 5 for the control group.

Samples underwent mechanical testing to compare the porous and contaminated samples against the controls. There was a significant difference in the mean ultimate tensile strength between the groups (p<0.0001, One-way ANOVA). Ultimate tensile strength of both fault conditions was shown to be significantly lower than the controls, Figure 21 (porous p<0.01, contaminated p<0.001, One-way ANOVA post hoc Bonferroni test). Elongation at break was also significant different between the groups (p<0.0001, One-way ANOVA) and the control group elongation was shown to be significantly greater than both fault conditions, Figure 21B (porous p<0.01, contaminated p<0.001, One-way ANOVA post hoc Bonferroni test). This supports the hypothesis that defects impact weld performance and that the samples under analysis represent
conditions would be undesirable for manufacturers and could result in unacceptable catheter performance depending on the application.

Figure 21: (A) Ultimate Tensile Strength Across Groups difference in means ($P<0.0001$). (B) difference in means of elongation at break ($P<0.0001$). **$P<0.01$, *** $P<0.001$ One-way ANOVA with a Bonferroni Post Test.
4.3 Fault Detection

As the samples created represented unwanted manufacturing faults, we examined the ability for both the Micro-CT and the created US setup to detect those faults in a population. Both the Micro-CT and US results presented below were performed on the same sample groups, prior to the destructive performance testing.

4.3.1. Micro-CT

To examine the total weld volume within the Micro-CT field of view, total material volume was examined across groups, and there was a significant difference in the mean volume between the groups (p<0.01, One-way ANOVA). Figure 22 shows that there was more material present across the weld area in the contaminated group as is expected from addition of foreign material to these samples during their creation. Though the field of view examined was held consistent across samples (3.99mm), there exists sample variance. Therefore, rather than examining the total volume of the porosity or debris detected, the percentage of the contaminants relative to the total weld volume was examined.

![Figure 22: Total Weld Volume Across Sample Groups difference in means (P=0.0064) *P<0.05, **P<0.01, One-way ANOVA with a Bonferroni Post Test.](image-url)
Porosity was defined as enclosed gaps or holes within the weld OD not including the ID of the tube where no polymer was present. Whereas contamination was defined as foreign matter with a higher density than that of the Pebax® material. The thresholding levels were held constant across groups.

The porous samples displayed a large variance in void percentage, volume of porosity relative to volume of welded region, within the group. This indicates that the sample creation method employed for the creation of the porous samples was inconsistent. The contaminated samples contained by far the largest debris percentage and there was significant difference between the groups (p<0.0001 One-way ANOVA). This indicates that within the porous group, the porosity was inconsistent, and this could contribute to varied impact on weld performance as seen in Figure 21.

**Figure 23:** (A) Void percentage across groups no difference in means (P=0.2077) (B) Debris percentage across groups difference in means (P<0.0001) ***P<0.001 One-way ANOVA with a Bonferroni Post Test.

Using the Micro-CT results, we investigated correlation between the total inclusion percentage (voids and debris) and ultimate tensile strength for all samples (Figure 24). Although the ultimate
tensile strength decreased with increasing total inclusion percentage ($p = 0.0809$), the correlation was weak ($r^2=0.19$). A cut off value to distinguish acceptable welds from rejects, is shown and positioned three standard deviations from the mean inclusion percentage of the controls. This results in a separation value of 0.87 indicating that not all faulty samples would have been detected using this inspection approach. Similar results were seen for the elongation at break data (Appendix: Supplementary Results).

![Figure 24: Correlation between total inclusion percentage and ultimate tensile strength (not significant, $P = 0.0809$, $r^2 = 0.19$). Cut off (red dotted line) value at 0.16. Separation value of 0.87.](image)

Examining the fault types separately (Figure 25), there was no significant correlation between void percentage and ultimate tensile strength ($P = 0.076$) while the correlation between void percentage and strain at break was significant ($P = 0.042$), but weakly correlated ($r^2= 0.3845$).
The correlation between debris percentage and ultimate tensile strength was found to be highly significant ($P<0.0001$) and a strong predictor with an $r^2$ of 0.9077 (Figure 26).

One outlier is seen when using debris as a predictor for tensile strength of the contaminated samples. A possible explanation is that the debris is highly concentrated at the weld interface,
when compared to the other contaminated samples. This would cause less material mixing during welding resulting in stress concentration across a smaller cross-sectional area. To check debris concentration, an upper limit of debris was calculated as twice the maximum debris percent per slice (approximately 0.3% of sample cross-sectional area). The outlier (contaminated sample #5) displayed the thinnest debris width at 0.180 mm. The next thinnest contaminated defect had a debris width of 0.267 mm. This would indicate that not only the total quantity of the contamination impacts weld performance but also the geometrical distribution of the contamination. Further exploration of the porosity morphology could provide more insight to weld performance.

Porosity and contamination within welds were shown to reduce the mechanical properties of the welds examined. There was significant correlation between the debris percentage and a decrease in ultimate tensile strength and elongation at break, but void percentage was only weakly predictive of ultimate tensile strength and elongation at break.

4.3.1. Normal Incidence US

B-mode US traces converted to c-scan images were used to determine if faults could be observed visually within the welds. It was assumed that if the defect size was larger than the axial resolution (0.13mm) and the step size (0.1mm), then it should be visible. This evaluation mode was limited to a single plane along the welded tube, and thus limited in scope.

The initial assumption held true for large defects such as bubbles (Figure 27A) though contamination defects were not observed as readily (Figure 27B). From the Micro-CT data, the debris was found to be concentrated within a range from 0.55 – 0.18 mm. However, as the contamination plane was inline with the ultrasonic wave propagation direction, the ability for the US to detect the fault is decreased as no distinct fault interface is present and detection would depend on the lateral resolution (beam diameter of 0.2mm) which is less sensitive. The 15MHz transducer used had a beam diameter of approximately 0.2mm at its focal point of 19mm, and the step size used was 0.1mm. Scanning time was 7 minutes to scan 9mm. The length of time required, and the limited resolution of in-plane defects makes this type of scanning impractical.
4.3.2. Angled Beam US

To minimize the required scanning time and improve defect detection the transducer was held stationary, and the test piece was rotated. The transducer was focused on the weld interface. The rotation allowed for the entire weld surface to be scanned. It was hypothesised that this technique would provide more information on the total inclusions across the whole weld rather than just a single plane. A total of 24 15° steps required 3 minutes to complete per sample. Additionally, the incident angle of faults at the weld interface should improve overall fault detection.

The initial hypothesis was that a continuous weld, would produce less reflected total energy than welds containing defects. The discontinuities in the porous and void samples should cause a much greater reflected signal energy. To test the hypothesis, total energy was compared across sample groups (Table 7). The control responses were in line with the hypothesized results returning a much lower total energy. Additionally, the variance within the control group was much smaller, indicating homogenous samples. The defect groups had a higher average mean total energy and much larger variance as expected. The mean total energy across groups was significantly different.
(p = 0.0059, Figure 28). The difference between the contaminated samples and the control samples was not statistically significant, as indicated by the post test.

Table 8: Total Normalized Energy Across Groups

<table>
<thead>
<tr>
<th>Group</th>
<th>n</th>
<th>Mean Energy ± Std.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>5</td>
<td>0.14 ± 0.06</td>
</tr>
<tr>
<td>Porous</td>
<td>6</td>
<td>1.93 ± 0.87</td>
</tr>
<tr>
<td>Contaminated</td>
<td>6</td>
<td>1.45 ± 0.84</td>
</tr>
</tbody>
</table>

Figure 28: Total normalized energy across groups, significant difference in means (P=0.0059) **P<0.01, One-way ANOVA with a Bonferroni Post Test.
Figure 29: Comparison of Micro-CT and US evaluations via total Inclusion Percentage versus Total energy response post bandpass filtering (2.5-30MHz). Contaminated sample 5 (red) energy did not increase with increased total inclusion percentage.

Figure 29 shows the Micro-CT total inclusion percentage versus the total energy. Fault groups are identified individually but evaluated as a population. For all fault samples (except Contaminated 5, Figure 29, red) as the inclusion percentage increases the total energy does as well. In general, the trend supports the hypothesis that a larger percentage of inclusions within a weld, results in higher total reflected energy however there likely exist limitations in the current US setup. The distinction between fault groups is evident, suggesting fault type also plays a role in US total energy response. Possible inclusion detection limitations could be linked to the transducer resolution if defects are concentrated and unable to be detected. Contaminated sample 5 had its debris concentrated across the thinnest width (0.180 mm), which approaches the transducer theoretical axial resolution in Pebax® 72D (0.132 mm). Contaminated sample 6 had the next thinnest debris concentration (0.268 mm). Additionally, the positioning and alignment of the samples relative to the transducer could impact results.

4.3.3. US Fault Detection

As the aim of the US NDE is to detect defects that could ultimately impact weld performance, the US signals were examined to determine if a possible correlation exists between the US energy and ultimate tensile strength.
For the subsequent analysis, contaminated sample 5 was not included and assumed that the defect concentration was below the limit of resolution of the transducer. Cut off values were calculated as 3 standard deviations away from the average response from the control group. The separation value was then calculated by comparing the lowest total energy from the fault groups to the cut off. Total energy calculation (Bandpass filter between 2.5MHz and 30MHz) resulted in a separation value of 1.3 (Figure 30). Frequency ranges were explored in 5 MHz step sizes across the transducer frequency spectrum (bandpass, low pas, and high pass). As initially expected, filtering at the lower frequency, larger wavelengths, range resulted in a larger separation value of 2.1 (Figure 31). These lower frequencies have a sensitivity to larger defects only.

**Figure 30**: Correlation between total normalized energy response post bandpass filtering (2.5-30MHz) and ultimate tensile strength (P= 0.0101, $R^2 = 0.3869$). Control group average 0.14±0.06. Cut off (red dotted line) at 0.33. Separation value of 1.3.
It was theorized that the weld performance could be largely dictated by large inclusions in this area, as there would be decreased material mixing resulting in higher stress concentration. It was thought that the largest inclusions would additionally produce the largest reflected energy. To
test this hypothesis, rather than the total energy response across the entire weld face, only the rotational step with the highest energy was compared (maximum single rf trace response). Across all filtering frequencies, the separation ratio was below 1, indicating that at least one defective sample would have not been detected. Though there was a significant difference in the means (p = 0.0204 One-Way ANOVA Figure 33), this single trace energy approach did not yield a greater separation value (i.e., did not improve distinction between groups). The highest separation value found was 0.9 and shown in Figure 34. Examining just the porous cases, a separation value of up to 1.5. Additionally, looking solely at the highest amplitude response (single point along trace), yielded similar results and did not improve defect detection.

![Figure 33: Mean difference across groups maximum normalized single trace energy post 5-15MHz bandpass filtering (P=0.0204)](image)

*Ps<0.05 One-way ANOVA with a Bonferroni Post Test.
Figure 34: (A) Normalized maximum single trace energy response post bandpass filtering (5-15MHz) versus ultimate tensile strength. No significant correlation. Control group average maximum single trace energy 0.27±0.0.10. Cut off (red dotted line) at 0.57. Separation value of 0.9. (B) Normalized maximum single trace energy response post bandpass filtering (5-15 MHz) versus elongation at break. Weak correlation (P=0.0295, r² = 0.2957). Control group average maximum single trace energy 0.27±0.0.10. Cut off (red dotted line) at 0.57. Separation value of 0.9.

The ideal scenario would be to have one such limit for multiple defect types, but as seen during the Micro-CT results, the type of defect has a greater impact on performance than simply total inclusion percentage. Subsequent analysis looked specifically at ways to identify compromised welds if a manufacturer was most interested in monitoring for air bubbles (porous) or debris (contamination).

Comparing the control and the porous samples a separation value of 2.9 was found when filtering using a bandpass filter between 5-15MHz. This large separation value indicates that it would be feasible to detect nonconforming welds when porosity is present. There was a significant correlation (p=0.0025, r² of 0.6551) demonstrating lower ultimate tensile strength samples produce increased reflected US energy (Figure 35A).
**Figure 35:** (A) Correlation between normalized energy response of porous and controls post bandpass filtering (5-15MHz) versus ultimate tensile strength ($P=0.0025$, $r^2=0.6551$). Control group average normalized energy $0.38\pm0.17$. Cut off (red dotted line) at 0.88. Separation value of 2.9. (B) Correlation between energy response of porous and controls post bandpass filtering (5-15MHz) versus strain at break ($P=0.0002$, $r^2 = 0.7925$). Control group average normalized energy $0.38\pm0.17$. Cut off (red dotted line) at 0.88. Separation value of 2.9.

Examining the contaminated samples, using a bandpass filter between 5-10 MHz resulted in the largest separation value of 2.1. As total energy increased, both ultimate tensile strength and strain at break decreased. With $r^2$ values of 0.7698 and 0.7091 respectively, a moderate predictive linear model could be established.
Mechanical properties of the welds were shown to decrease with increased signal energy response during angled beam US inspection. A potential limit of resolution was identified for this setup approach. A separation value, used to distinguish the acceptable and unacceptable welds, was established across the population of up to 2.1. There was a significant correlation between the porosity energy response and a decrease in ultimate tensile strength and elongation at break. A separation value of up to 2.9 was shown when inspecting samples specifically for porosity.
5. DISCUSSION

This thesis demonstrated that a low-cost US setup can identify defects in catheter welds. The results show that the energy reflected is indicative of weld quality and that a pass/fail criteria can be established and identify welds that contained defects that impacted the mechanical properties of the weld.

5.1. Morphology and Weld Integrity

In order to inspect the catheter welds, a custom inspection setup was created. This setup allowed for normal and angled US NDE along the length of the weld and rotationally. The scanning time for a single normal inspection was 7 minutes, which would not be feasible to implement on a manufacturing line as multiple normal scans would be required to fully inspect a weld. Scanning time was greatly improved using the angled beam approach requiring just 3 minutes per sample. A single weld step during manufacturing takes between 1-3 minutes. An additional limitation to manufacturing use is the need for a coupling medium. This feasibility approach used water, which is not compatible for use in a cleanroom environment. However, possible alternatives could be used such as an alcohol, which is often used to disinfect and clean components during assembly. Other groups have explored other coupling methods such as water droplets [23], low loss silicone rubber [42], or air-coupling [43]. The set up used was limited by the resolution of the prototype parts. Many components were 3D printed where the tolerancing was limited (~0.2 mm). Deviation in concentricity could lead to inaccurate results during angled beam inspection, while alignment of components could impact normal imaging. This limitation could be overcome by using machined parts, leading to improved repeatability and reliability. However, with the experimental limitations outlined above, the setup was capable of measuring catheter wall thickness using TOF methods to 7.8% of nominal tube wall thickness when compared to Micro-CT measurements (RMSE = 0.035mm), Figure 20. The current set up would be sufficient to validate catheter geometry when wall thickness is critical to catheter performance and patient safety.

Representative catheter weld samples were created using Pebax® 72D, an engineering thermoplastic (polyamide) which is biocompatible and is commonly used in medical devices
today. A single lumen 7.3Fr butt weld was examined, and the process was shown to be consistent and reliable across the control group, indicating that the process was under control (mean tensile strength of 84.0±1.7N, mean elongation at break of 432.3±28.6%). Industry commonly uses this type of weld during construction of balloon catheters scaled to the specific application (3-34Fr).

To examine the impact of faults on weld performance, porosity and contamination was introduced within the weld. Both these types of subsurface defects would not be detectable via visual inspection in coloured or opaque materials. These samples were created with non-graded defect sizing leading to variability across samples. The presence of these defects was shown to produce a significant negative impact on ultimate tensile strength and elongation at break (Figure 21). Contaminated samples displayed a more severe reduction in ultimate tensile strength with almost no elongation. The ultimate tensile strength and elongation at break were much more variable in the porous samples. A high variation in void percentage was present across the porous sample group. Though the ultimate tensile strength was above the minimum requirement for a weld diameter of this size (15N [31]), performance requirements now mandate that acceptance criteria be based on clinical application and intended use [44]. Thus, any decrease in weld performance may impact the safety profile of the device depending on intended use and both types of defects created would lead to an unacceptable product. The manufactured defects in this thesis would likely be rare in a certified production process. However, if such defects did occur, they may not be detected as validation relies on statistical sampling rather than 100 percent verification. Therefore, the faults seen are representative of defects that could ultimately compromise patient safety if they were to leave the manufacturing floor. Welds that do not meet performance requirements could result in component detachment and entrapment (i.e., during catheter withdrawal) requiring emergent surgery for removal.
5.2 Micro-CT Fault Detection

Micro-CT was used as a means of US calibration and volumetric fault analysis as is common due to its high resolution and repeatability [18, 45]. The total weld volume analysed using the Micro-CT was significantly different across groups (Figure 22) and subsequent analysis was performed relative to the specific sample weld volume. The contaminated samples were shown to include a significantly larger mean volume than the other two groups. This is inline with sample creation, as foreign material was added to the weld. In Figure 23, types of inclusions were examined across groups. The void percentage was shown to be varied across porous samples and potentially dispersed throughout the weld by the pressure applied by the outer jacket during welding. Whereas the debris within the contaminated samples would likely be much more localized to the weld face and not as likely to mix within the liquefied polymer during welding. The contamination was shown to be limited to 0.18-0.55mm in width.

It was hypothesized that the presence of defects would decrease weld performance and that a higher quantity of defects would lead to greater decreases in weld performance. This was tested examining the correlation of total inclusion percentage to ultimate tensile strength (Figure 24). Though ultimate tensile strength decreased with increasing inclusion percentage (p=0.0809) the correlation was weak (r^2=0.19). This weak relationship is potentially caused by the different fault types and the assumption that porosity and contamination could be considered a continuum of defects does not stand. Attempts to create a pass/fail cut off using total inclusion percentage was unsuccessful and the separation value was < 1.

Looking at the void percentage as a predictor of weld performance (Figure 25) a weak correlation was seen (p=0.076). Performance was shown to decrease with the presence of voids within the welds, however the greatest quantity of voids did not equate to the lowest tensile strength. This could indicate that the concentration of the voids also plays a role in weld performance. Higher concentration of voids could decrease cross sectional weld area and create highly concentrated stress locations.

Examining the debris percentage as a predictor of weld performance yielded highly significant results (p<0.0001) with a strong linear relationship (r^2 = 0.9077), Figure 26. One outlier was
observed, and it was examined for debris concentration and displayed the thinnest debris width (0.18mm). This could have led to limited material mixing during welding when compared to the other samples.

Additional investigations in the morphology and concentration of the porosity and debris across the weld area could be performed through 3D analysis using Micro-CT offline processing. However, it would not necessarily be predictive of mechanical properties of the weld as the porosity results show. Even with the additional morphology information, the limitations of Micro-CT, such as scanning and processing time and equipment cost, prevent its use for inline manufacturing of many catheter devices. In this work, Micro-CT scanning took approximately 40 minutes per sample, with an additional hour in offline processing. The offline processing time could be reduced with the use of improved processing power however the scan time makes it impractical for use in an endovascular device manufacturing setting. Comparatively, the angled US approach took approximately 3 minutes to scan and seconds for offline processing.
5.3. US Fault Detection

Normal US (B-mode imaging) was used to visualize the samples and to test if fault could be observed within the welds. Porous samples were readily observed however, as the scan consisted of a single plane the value for predicting weld performance is limited. Additionally, defects parallel to the incident US plane were not readily detected and these types of faults were shown to greatly decrease weld performance. As such angled beam US evaluation methods were used.

Angled beam US was employed to decrease required scanning time, by focusing on the critical weld interface along its entire length. It was hypothesized that homogenous welds would reflect significantly less energy when compared to welds containing defects. When comparing the total energy between the samples, a significant difference in means across the groups was evident (P=0.0059 One-Way ANOVA), Figure 28. When the total energy was plotted with respect to the CT inclusion percentage, Figure 29, a distinction between fault types can be observed along with outliers in each group. The contaminated outlier (Figure 29 red), was the sample with the thinnest debris width and the lowest inclusion percentage. This could indicate that the sample defects were below the US limit of detection or that experimental setup error (alignment or positioning) contributed to this result. This detection limit contributed to the non-significant difference in means between the control and contaminated samples shown in Figure 28. Removal of that sample results in a significant difference in means between the control and contaminated sample groups (P<0.01). It was assumed that the detection limit for angle beam US was between 0.18-0.27mm, and this sample was removed for further analysis.

Excluding contaminated sample 5 (below limit of detection), a cut off value was established that can differential faulty welds from the control group. The sensitivity of this measurement was estimated using a separation value. The greater the separation value, the lower the likelihood of error (i.e., false positive). Across the population, a normalized energy cut off value of 0.33 resulted in a separation value of 1.3 (Figure 30) and using additional filtering the separation value could be improved to 2.1 (Figure 32).

Similar to the Micro-CT results, looking for specific fault types yielded better results. This could be used by manufacturers to inspect for a known fault cause while controlling the process for
other defect types (i.e., porosity when samples are made in a controlled cleanroom environment). Using total energy as a predictor of ultimate tensile strength in porous samples, the correlation was found to be significant (p=0.0025), following a linear mode ($r^2=0.6551$). This relationship was found to be stronger than that of the benchmark Micro-CT. While inspecting specifically for contamination, a separation value of 2.1 could be established. The correlation between energy and ultimate tensile strength was found to be significant (p=0.0009) with a good linear model ($r^2=0.7698$). In both defect groups, increasing energy response was correlated with decreased mechanical properties of the welds.

As hypothesized, homogenous samples produced limited reflected energy when compared to faulty welds. Welds containing faults could be identified, except for one sample, using established cut off limits in energy response. This result supports the feasibility of US NDE to inspect catheter welds.
5.4 Limitations

Setup limitations were previously discussed, and the follow are limitations of the experimental design. The samples were not scaled or include graded defects to determine detection resolution, as in other studies [22], but were inspected retroactively using Micro-CT to quantify inclusion volumes. Study included a small sample size and examined the simplest endovascular catheter weld, a single tube geometry. Using verification through angle beam US, each type of weld would require baseline measurements to be performed to determine the cut off value for a specific weld. Image registration between the field of view in the Micro-CT and the US was not controlled.
6. CONCLUSION & SUMMARY

A bench top setup was created that was able to perform both multiangled US NDE on endovascular catheter welds for less than $7000 USD. The setup allowed for wall thickness measurements comparable to those performed using Micro-CT. The scanning time required per sample was using US was 3 minutes compared to the 40 minutes required for Micro-CT scanning. Samples were created that represented common welding faults seen in industry, porosity, and contamination, and was shown to have a negative impact on overall weld performance. Samples with faults displayed a decrease in ultimate tensile strength and elongation at break. The contaminated samples displayed the greatest decrease in both ultimate tensile strength and elongation at break.

Faults were volumetrically measured using Micro-CT and a general trend of decreased performance with increased inclusion percentage was shown. Examining overall fault detection of the Micro-CT, total inclusion percent did not display a significant correlation with decreased ultimate tensile strength and resulted in a separation value of 0.87. Overall fault detection of angle beam US displayed a significant correlation between increasing energy and decreasing ultimate tensile strength and had a separation value of up to 2.1 post filtering. However, one sample was not detected using the US setup which could indicate a contamination detection limit between 0.18-0.27 mm.

Examining for porosity specifically as a fault type, the US energy was a stronger predictor of decreased ultimate tensile strength through the energy response as compare to the void percentage measured by the Micro-CT ($r^2=0.6551$ and $r^2 = 0.3087$, respectively). Examining for contamination specifically as a fault type, the Micro-CT was a stronger predictor of decreased ultimate tensile strength via debris percentage as compared to US energy ($r^2 = 0.9077$ and $r^2 = 0.7698$, respectively).

This study demonstrates the feasibility of US NDE to monitor and verify the output of endovascular welds, which could reduce the burden of process validation for manufacturers.
7. FUTURE WORK

To augment the created setup, integration of multiple US scanning techniques (normal, angled) could improve fault detection and allow for simultaneous wall thickness measurement. Multielement transducers (phased arrays) could be explored to allow for weld interface scanning without requiring transducer displacement. Combinations of transducer frequencies could be employed to detect different fault types and potentially allow for fault identification through frequency analysis. Along with different transducer setups, the coupling agent could be explored specifically for methods that are compatible with cleanroom environments such as isopropyl alcohol, drip coupling, or low loss silicone rubbers.

Specific evaluations of fault morphologies could help to better predict individual fault type impacts on weld performance. Micro-CT could be used to determine porosity concentration, pore size distribution, and resultant minimum cross-sectional area across the weld interface.

This work examined the base case weld commonly found on balloon catheters; many more complex weld geometries could also be studied to support US NDE fault detection capabilities. Additionally, this work examined the response from a single material. Future work could examine welds featuring multiple materials and the interaction at the weld interface.
8. CONTRIBUTION OF AUTHORS

Experiments presented were designed in consultation with Richard Leask, Rosaire Mongrain, Paul Fromme and Robert Earl. Robert Earl created the setup for experiments, conducted the experiments, and collected the data. Interpretation of the data done in discussion Richard Leask, Paul Fromme, and Robert Earl. Robert Earl wrote the thesis and his supervisors, Richard Leask and Rosaire Mongrain helped revise.


44. Peripheral Percutaneous Transluminal Angioplasty (PTA) and Specialty Catheters - Premarket Notification (510(k)) Submissions Draft Guidance for Industry and Food and Drug Administration Staff. 2020, FDA - U.S. Department of Health and Human Services Food and Drug Administration Center for Devices and Radiological Health: USA.

10. APPENDIX: ADDITIONAL FIGURES

Figure 37: Rendering of angled beam ultrasound setup. Sample and transducer coupled via water.

Figure 38: Angled Beam US focusing using a half sample. Responses from the half sample was used for normalizing the energy values as resistance was unknown.
**Figure 39:** Example partial trace used for time of flight wall thickness calculations.

**Figure 40:** Olympus 15MHz transducer frequency spectrum
11. APPENDIX: SUPPLEMENTARY RESULTS

![Graph showing correlation between total inclusion percentage and elongation at break.](image)

**Figure 41:** Correlation between total inclusion percentage and elongation at break ($P = 0.0152$, $r^2 = 0.3334$). Cut off (red dotted line) value at 0.16. Separation value of 0.87.

12. APPENDIX: SOFTWARE

Software is freely available on request from richard.leask@mcgill.ca.