
Charles Landais

University of Nebraska-Lincoln, charles.landais@gmail.com

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ULTRASONIC METHODS FOR THE CHARACTERIZATION OF COMPLEX MATERIALS AND MATERIAL SYSTEMS: POLYMERS, STRUCTURED POLYMERS, SOFT TISSUE AND BONE

by

Charles Landais

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ULTRASONIC METHODS FOR THE CHARACTERIZATION OF COMPLEX MATERIALS AND MATERIAL SYSTEMS: POLYMERS, STRUCTURED POLYMERS, SOFT TISSUE AND BONE

Charles Landais, M.S.

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Advisers: Mehrdad Negahban and Joseph A. Turner

Understanding the characteristics and structure of materials is of importance in proper modeling and effective design of many products. Complex materials such as polymers, structured material systems, or biological materials provide a particular challenge to many of the traditional methods for doing this. In this dissertation I study the use of ultrasonic wave techniques to characterize several complex materials. These include plastically deformed and aged polycarbonate (PC), a structured PC plate with water filled cavities, bovine bone, and an elastomer used as a skin simulant. In each case, this work was part of a larger project that studied different aspects of related problems.

Ultrasonic methods use high frequency mechanical waves to interrogate the structure and properties of materials. As a result, ultrasonic waves are capable of characterizing the response of materials during high frequency loading and can characterize structure at fairly small sizes. As such, the ultrasonic techniques provide a special window of characterization that may provide important information on the response of complex materials such as polymers and structured material systems such as is seen in bone.
In the study of anisotropic ultrasonic waves in plastically worked and then thermally aged samples, it is shown that the large drop in toughness seen with aging does not correlate closely with the gradual changes observed in the ultrasonic wave speeds. In developing a structured polymer by producing different topologies of water-filled channels by rapid prototyping with a pseudo-PC material, it was shown that one can make plates with overall ultrasonic wave speeds between the speeds in pseudo-PC and water, with the response under some topologies showing close to linear interpolation between the two materials. In studying cow bone, we were able to evaluate the amplitude and attenuation using contact transducers in the pulse-echo mode, and this provided an estimate of the critical angle for shear wave formation in submerged ultrasonic in a water tank. In evaluating the viscoelastic properties through contact surface reflections, the properties of reference materials were evaluated using the standard pulse-echo method, but preliminary results of the contact-reflection method did not correlate with these results (see the master thesis of Jonathan Hein for follow-up work that was done to obtain good correlations).
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Chapter 1

INTRODUCTION

1.1 Introduction

Understanding the characteristics and structure of materials is of importance in proper modeling and effective design of many products. Complex materials such as polymers, structured material systems, or biological materials provide a particular challenge to many of the traditional methods for doing this. In this dissertation I study the use of ultrasonic wave techniques to characterize several complex materials and material systems. These include plastically deformed and aged polycarbonate (PC), a structured PC plate with water filled cavities, bovine bone, and a PDMS used as a skin simulant. In each case, this work was part of a larger project that studied different aspects of the problem.

In my studies I have used two ultrasonic setups. These include the contact transducer method and the fluid assisted ultrasonic technique in a fluid bath. A
description of ultrasound and the ultrasonic techniques and the basic method with contact transducers and in a water bath are described in following sections.

1.2 Ultrasound

Ultrasonic waves are mechanical waves that are commonly used in nondestructive testing applied to a wide variety of material analysis applications. For example, ultrasonic techniques are used for thickness gauging, flaw detection and acoustic imaging and can also be used to define and quantify mechanical properties such as density and elastic modulus, and to characterize internal structural features of parts. Typical measurement parameters are the time of flight, attenuation, scattering, and the change in the frequency spectrum of a wave in a test object, which, with mathematical modeling provide the means to obtain information about sample geometry and material properties [1].

Ultrasonic waves are defined as mechanical waves that propagate at frequencies that are above 20 kHz, i.e. they start from the upper limit of the audible range for humans. Ultrasonic tests can be performed with either longitudinal waves or transverse (shear) waves. Longitudinal waves are waves that have their displacement oscillations along the direction of the propagation of the wave. Transverse or shear waves are waves that use displacement oscillations that occur at a right angle (or transverse) to the direction of wave propagation. Solids typically support both longitudinal and shear waves, while fluids typically support longitudinal waves, but frequently have difficulty transmitting shear waves [2].

Piezoelectric devices are generally used to generate and sense ultrasonic waves. These transducers convert electrical energy into acoustical energy and vice-versa, and can
be used either as a transmitter or as a receiver. Different transducers are used for different testing conditions and materials. High frequency transducers (i.e. from 15 MHz to 25 MHz) provide reduced penetration into a material but a great sensitivity to small discontinuities. In contrast, low frequency transducers (i.e. from 0.5 MHz to 2.25MHz) provide a greater energy and penetration depth, but a reduced sensitivity to small discontinuities [2].

To setup an experiment several functional units are needed. These are typically a pulser/receiver, a transducer, and a display device. A pulser/receiver is an electronic device that can produce electrical pulses [1]. Driven by the pulser, the transducer generates high frequency ultrasonic oscillations. These oscillations when coming in contact to a sample produce waves at the surface of the sample that propagate into the material. In the case of the pulse echo method, when the wave is reflected at an interface, the signal travels back to the transducer and is detected and transformed into an electrical signal by the same transducer and the resulting electrical signal can be displayed on a screen. In the case of the through transmission method, the wave is received by a second transducer positioned on the other side of the sample, and then acquired, saved, and possibly displayed by an oscilloscope.

1.3 Contact ultrasonic method

The contact ultrasonic method uses a transducer that is placed directly in contact with the sample under consideration. For example, in a pulse-echo setup, the test is performed with a single transducer in contact with the test object. The transducer both generates and receives the signal. The generated ultrasound pulse goes through the sample and is
reflected at an interface (e.g. flaws or edges), and is detected by the same transducer as shown in Figure 1.

![Diagram](image)

**Figure 1**: Schematic representation of the contact ultrasonic setup for a pulse-echo method.

In contrast to the fluid assisted through transmission ultrasonic method in a bath, the quality of the signal for the contact method is closely connected to transducer-sample contact quality. Frequently there is a need for smooth surfaces, the aid of a couplant, and careful positioning to obtain good results. The main source of error in this method comes from poor contact between the transducer and the sample, which results from samples that are not flat, smooth or parallel [1].

1.4 Fluid assisted through transmission ultrasonic method

The fluid-assisted methods use a fluid medium between the transducer and the sample. This is normally realized by setting the sample in a water tank that includes submerged transducers. Two typical setups exist. In the first case the sample is fixed and the transducers moved by the aid of a turntable. In the second case the transducers are fixed and the sample is moved.

For this technique at least two transducers are needed. One transducer acts as a transmitter while the second acts as receiver. To receive all the ultrasonic energy sent by the transmitter and passed through the sample the two transducers have to be well
aligned. Two holders are used to control this alignment. As for the pulse-echo method, a change in the signal at the second transducer is related to the material [1]. A schematic representation of the through transmission method is given in Figure 2.

![Figure 2](image)

**Figure 2:** Principle of the through transmission ultrasonic method in a water bath.

In contrast with the contact measurements, this method allows the sample to be oriented relative to the transducers so that the wave can be made to impact the surface of the sample at different angles and at different locations. This can be controlled automatically through a test positioning and rotation system. For example, the incident angle can be controlled by putting the sample or the transducers on a turntable. Because the setup is submerged in a water bath, the immersion technique also provides a continuous fluid medium between the transducers and the test object thereby circumventing the problems of coupling of transducer and sample, which can occur while using contact transducers such as in the pulse-echo method, for example. In addition to allowing faster measurements, compared to the contact method, the immersion technique is not as much influenced by loss of coupling due to ovality of tubing, surface conditions or dimensional variations. For materials with high attenuation such as bone, the through transmission method is generally preferred since the waves only go through the material.
once [1]. Because the samples are immersed into a water bath, the use of samples susceptible to corrosion is limited. Because of the weight and the numerous devices need for the measurements, this system suffers from a lack of mobility. In order to limit the bubbles, that can introduce inaccuracy in the measurements, the tank needs to be filled with water two or three days before the experiments [1].

1.5 Ultrasonic data processing

From ultrasonic tests several relevant parameters such as the wave speed and the attenuation are deduced. These parameters are generally used to define and characterize a material.

The wave speed is usually the easiest ultrasonic parameter to measure. The speed of sound in a homogenous medium is directly related to both the elastic modulus and density. Thus, changes in either elasticity or density will affect the pulse travel time through a sample of a given thickness. Additionally, varying degrees of nonhomogeneity may have an effect on sound velocity.

The principle of a pulse-echo contact ultrasonic testing measurement is represented in Figure 3. In this case the reflected signal strength can be displayed versus time for both the signal generation and the echo received by using an oscilloscope. The signal travel time can be directly related to the distance that the signal traveled giving information such as flaw sizes and location.
Figure 3: Principle of the pulse echo method.

For a signal pulse that propagates through a sample with a thickness $d$ and that is reflected at the opposite wall of the sample and produces echoes, each echo travels a distance equal to twice the sample thickness $d$ before it reaches the transducer again. By measuring the time delay $t$ between two echoes, the ultrasonic velocity $v$ can be calculated using

$$v = \frac{2d}{t}.$$  

(1)

The longitudinal wave modulus $E$ is calculated by

$$E = \rho v^2,$$  

(2)

where $\rho$ is the density. The density can be measured with a scale using

$$\rho = \frac{A}{A-B} \rho_0,$$  

(3)

where $A$ is the weight of the solid in air, $B$ is the weight of the solid in an auxiliary liquid of know density such as distilled water, and $\rho_0$ is the density of the auxiliary liquid at the given temperature. Distilled water is often used as the reference fluid. At $22.5^\circ C$ the
density of distilled water is 0.99768 g.cm$^3$. The measurements were done using a Metler Toledo AT201 scale.

When a wave travels through a medium, its intensity decreases with distance. This decrease is called attenuation and refers to two main phenomena: scattering and absorption. Scattering can be defined as the reflection of the sound in directions other than its original direction of propagation while absorption is the conversion of the sound energy to other forms of energy, such as heat.

Ultrasonic measurements provide information related to the ultrasonic attenuation. Sound attenuation is related to the sound energy that is absorbed or attenuated in a material. This is governed in a complex way by interactive effects of density, hardness, viscosity, and molecular structure. Attenuation normally increases with frequency in a given material.

Ultrasonic attenuation can be used as a qualitative value to infer properties of the solid [1]. Exponential attenuation can be expressed by

$$ A = A_0 e^{-\alpha x}, \quad \text{(4)} $$

where $\alpha$ is the attenuation coefficient of the wave and is expressed in Nepers per length, $A_0$ is the unattenuated amplitude of the propagating wave. The amplitude $A$ is the reduced amplitude after the wave has traveled a distance $x$ from that initial location.

The value of the attenuation coefficient depends on the frequency of the signal and is normally reported for a given frequency or as an average for a particular frequency range. The attenuation also is effected by the manufacturing process, so the same material
might show different attenuation depending on the manufacturing method and processing conditions.

The attenuation coefficient and wave speeds are functions of the frequency. To help analyze this dependence, the fast Fourier transform (FFT) is used on the signals to separate it into its different components. This is because the ultrasonic signals normally include multiple frequencies. Fast Fourier Transform is a faster version of the Discrete Fourier Transform (DFT). By applying the FFT, a time domain signal will be expressed in the frequency domain in terms of the decomposition of a pulse into a distribution of amplitudes for sinusoids of different frequencies. A time domain pulse is transformed into a frequency domain through the Fourier transform defined as

\[
F(\omega) = \int_{-\infty}^{\infty} f(t)e^{i\omega t} dt,
\]

where \( F(\omega) \) is the Fourier transform of \( f(t) \) and \( i = \sqrt{-1} \) [3]. The Matlab program and OriginPro were used to process the FFT during this study.
Chapter 2

PLASTIC DEFORMATION AND AGING OF PC

2.1 Introduction

Many polymers can be toughened by plastic deformation [4]. As part of an effort to study the effect of thermal aging on plastic flow induced toughening, the change of wave speed in samples of polycarbonate (PC) was studied. In this case samples were plastically compressed and then thermally aged by exposing them to different aging temperatures and aging times.

The effect of mechanical toughening (by plastic flow) in glassy polymers is very large so that any process that can reduce this effect is of importance. In our study, we had samples subjected to up to 50% plastic compression that showed up 15 times higher toughness in Charpy tests. After the studying of the effect of thermal aging on toughened samples, we noticed that the large increases in toughness measured by Charpy tests in some samples could be completely lost after thermal aging. This drop was seen to happen at different extents of aging for samples cut along different direction relative to the
compression (in this case the same amount of compression was applied but the sample orientation to the compression was changed).

The goal of the ultrasonic study was to determine if there is a correlation between the observed 10 to 15 fold drop in Charpy test results and ultrasonic measurements. If this would be the case, ultrasonic measurements could be used to evaluate the loss in toughness as a result of thermal aging. The results of the ultrasonic tests indicated that there is some correlation, but that ultrasonic measurements alone cannot be used to determine the loss in toughness.

This chapter starts with a description of PC, followed by a description of the plastic working and aging for the sample tested, and a description of the sample geometry and orientation relative to the deformation. The chapter concludes with the ultrasonic results, and a discussion of the transition seen in the Charpy tests and its comparison to the ultrasonic test results.

2.2 Introduction to polycarbonate

Polymers can be separated into three categories: thermosets, elastomers, and thermoplastics. Polycarbonate is a linear thermoplastic. The molecular chains that compose thermoplastics are not crosslinked or connected allowing, under the effect of a force and/or heating above the glass transition temperature ($T_g$), their molecular chains to slide over each other. The sliding around of these polymer chains is what allows thermoplastics to flow when heated or when loaded excessively [5].
Polycarbonate is used in many commercial products such as electronics, food packaging, and it is used for medical and optical applications [6] due to its good mechanical properties, its high impact resistance, and its good temperature resistance.

All experiments in this study were performed on a PC called Lexan 9034 and provided by the company Regal Plastics. This polycarbonate is formed with the reaction of bisphenol A (BPA) (produced through the condensation of phenol with acetone under acidic conditions) with carbonyl chloride. The polycarbonate structure resulting from this reaction is shown in Figure 4.

![Chemical structure of Lexan polycarbonate](image)

**Figure 4:** Chemical structure of Lexan polycarbonate [7].

Aging leads to a change in the polycarbonate properties. In fact, thermal history influences its properties. There are two kinds of aging: physical aging and chemical aging. In the first case the chemical structure of macromolecules is not changed, only its spatial configuration or material composition will change [8][9], and this results in a change in the material properties with time [10]. While in the case of chemical aging the structure of the macromolecules will be changed.

In this study we will consider physical aging mainly caused by the structural relaxation. The choice of aging temperature is critical because it must be low enough to limit the molecular motion, but large enough to allow relaxation enthalpy to achieve
equilibrium in reasonable times, usually it is around 120° C for polycarbonate, which has a glass transition of 147° C [9][11].

The properties of PC also depend on its mechanical history. Indeed, when subjected to loads which lead to a plastic deformation, an anisotropic behavior in PC is observed [12]. This behavior has been studied by Goel & al. [12] using both the modeling of anisotropy in PC due to compression and its measurement by using ultrasonic wave speeds [13].

2.3 Experiments conducted

The primary purpose of this study is to determine the effect of physical aging on the fracture toughness of plastically deformed PC. The ultrasonic tests performed were in support of this study and to measure the change in longitudinal wave speeds and the longitudinal wave moduli resulting from physical aging.

Experiments were performed on PC samples with a plastic compression of 0%, 25% and 50% strain for two temperatures of physical aging of 105° C and 125° C, and for various amounts of aging time up to 6000 hours.

2.4 Sample preparation

Different steps are necessary to obtain the final samples. The first step was the compression of a PC sheet by 25% or 50% strain. Then, the PC sheet was aged for various times from 0 to 6000 hours. PC samples, cut out from sheet, were prepared by Kyle Strabala and Shawn Meagher for Charpy testing and after completion of these tests they were used for ultrasonic evaluations. The samples were prepared with TT and TA
designations. As seen in Figure 5, the TT Charpy samples had a long side perpendicular to the axis of compression and were cut and notched such that the crack would run transverse to the compression axis. The TA Charpy samples had a long side that was perpendicular to the axis of compression, but were cut and notched such that the crack would run along the axis of compression. Since the ultrasonic tests were done along the thickness direction of the sample, the TT samples resulted in axial waves and wave speeds, and the TA samples resulted in transverse wave and wave speeds. Before being tested samples were slightly polished to decrease the roughness of their surface to obtain a flat area for measurements.

Figure 5: Labeling method for the orientation of cut samples [14].
2.5 Ultrasonic tests

The ultrasonic tests were performed using a square wave generator (Olympus 5077PR) and a 1MHz longitudinal contact transducer (Panametrics V103). The pulse-echo method was used to calculate the longitudinal wave speed by recording the time of flight between the second and third reflection. The signals were recorded using a Labview program running on a PXI expansion bus (National Instruments).

2.6 Ultrasonic test results

This study was done in parallel to a study on the fracture of deformed and aged PC samples. In order to compare these results, samples with various amounts of plastic compression followed by physical aging were prepared and tested. At 105° C aging temperature, the plastic compressions ranged from uncompressed to approximately 50% plastic engineering strain in compression and the physical aging ranged from 300 to 6000 hours. For physical aging at 125° C, the plastic strains ranged from zero to 50% engineering strain and the physical aging time ranged from zero to 600 hours.

The results for physical aging at 105° C are presented in Figure 6. As can be seen, under all conditions of plastic compression and aging the wave speed (and modulus) are higher for the TA samples than for the TT samples. It means that the transverse modulus is larger than the axial modulus. In addition, for the TA samples the compression increases the wave modulus while for the TT samples the compression leads to a decrease of the wave modulus. For the TT samples, there was an effect from aging on the wave speeds and associated moduli but this change was in most cases within the uncertainty of the results. For the TA samples a large decrease in the wave moduli between 0 and 300
hours of aging is observed, after this aging time the variations of the wave moduli are within the uncertainty of the results.

**Figure 6:** Axial wave modulus for PC samples aged at 105°C.

The results for physical aging at 125°C are presented Figure 7. As can be seen, there is a difference between the 25% and 50% compressed samples. Under all conditions of plastic compression and aging the wave speed (and modulus) are higher for the TA samples than for the TT samples. This means that the transverse modulus is larger than the axial modulus. For the TA samples, both the 25% and the 50% compressed samples show within 20 hours of aging a significant drop in the wave modulus. However, for the 25% compressed samples a jump at 50 hours of aging was observed. A similar behavior was also noticed for the initial uncompressed and unaged samples with a jump at 50 hours of aging. For the 50% compressed TT samples, there was an influence of aging on
the wave speeds and associated moduli but this change was in most cases within the uncertainty of the results. In contrast, the 25% compressed TA samples showed between 10 and 20 hours of aging a drop in the wave modulus.

![Figure 7: Axial wave modulus for PC samples aged at 125° C.](image)

2.7 Comparison between ultrasonic and Charpy results

The results of Charpy tests for a physical aging at 105° C are presented in Figure 8. As can be seen, under all conditions of plastic compression and aging the fracture energy is higher for the TA samples than for the TT samples. The initial uncompressed and unaged samples fail in a brittle manner and have a relatively low toughness. For the 25% compressed TA samples, a significant drop in the fracture energy between 3000 and 6000 hours of aging was observed. By contrast, the 50% compressed TA samples show a higher initial toughness and there was no loss in the fracture energy with time. For the
25% compressed TT samples, a significant drop in the fracture energy between 300 and 700 hours of aging was observed. The samples become brittle and the toughness decreases to approximately that of the undeformed samples.

![Graph showing fracture energy over time for different compressed samples](image)

**Figure 8**: Charpy test results for PC samples aged at 105° C [14].

The results of the Charpy tests that have been done on PC samples at an aging temperature of 125° C are reported Figure 9. The initial uncompressed and unaged samples fail in a brittle manner and have a relatively low toughness. For the TA samples, the 25% compressed samples show a lower toughness than the 50% compressed samples and with 20 hours of aging the samples become brittle and their toughness decreases to be approximately at the same level as the uncompressed and unaged samples. This loss of toughness was not observed for the 50% compressed TA samples. For the TT samples, the initial toughness of the 25% compressed samples is higher than the 50% compressed
samples but within 10 hours of aging the samples became brittle and their toughness dropped down to approximately the toughness of the uncompressed and unaged samples. As for the 50% compressed TA sample, the 50% compressed TT samples did not show a change in their behavior.

![Figure 9: Charpy test results for PC samples aged at 125° C [14].](image)

2.8 Conclusion

Ultrasonic tests were performed on PC samples that had various amounts of plastic compression followed by physical aging. At 105° C aging temperature, the plastic compressions ranged from uncompressed to approximately 50% plastic engineering strain in compression and the physical aging ranged from 300 to 6000 hours. For samples physically aging at 125° C, the plastic strains ranged from zero to 50% engineering strain and the physical aging time ranged from zero to 600 hours.
In regard to the wave modulus and the fracture energy, both the ultrasonic tests and the Charpy tests indicate that there is a difference between the 25% and 50% compressed samples whatever the aging time.

For the 25% compressed samples aged at 105° C two noticeable drops in the fracture energy were observed, one between 300 and 700 hours of aging for the TT samples and one between 3000 and 6000 hours of aging for the TA samples. Similar changes in the material behavior were not observed with regard to the ultrasonic test results. For this aging temperature, the ultrasonic tests do not show the ductile to brittle transition, while a substantial drop was observed with Charpy tests. In addition, the decreases of wave moduli, observed for the 25% and 50% TA samples between 0 and 300 hours of aging, with the ultrasonic tests were not observed in the Charpy results.

For the 125° C aging temperature a significant change was observed for the 25% compressed sample. For the 25% TT samples a 10 fold drop was observed in the Charpy results in the first 20 hours of aging. This drop seems to correlate with the drop observed in the axial wave speed in this same region. There was no other significant drop in the ultrasonic results that could be correlated with the observed drop in the Charpy result in the TA samples. However, there was a gradual transition in the ultrasonic results, and an unexplained bump which occurred in the undeformed and 25% compressed samples at about 80-90 hours. This could indicate a processing induced effect that existed in all samples, but which was depressed with the 50% compression.

The comparison between the Charpy tests and the ultrasonic measurements highlight that there is a possible correlation between the results, but it also shows that the
use of ultrasound does not allow us to determine as accurate correlation to the Charpy toughness tests.
Chapter 3

STRUCTURED PSEUDO-POLYCARBONATE

3.1 Introduction

In constructing models made of synthetic materials one issue is the selection of a material that can produce similar behavior as the original. It turns out that finding similar synthetic materials to reproduce the behavior of bone is difficult. One possible way to control the material response is to construct it with a controllable microstructure so that the composite system has closer behavior to the desired response.

In an effort to study the possibility of using this idea to control the response, we used rapid prototyping to construct samples that contain channels that were then filled with water. The samples were made of a material similar to polycarbonate (here it will be called pseudo-PC), and channels with different sizes, densities and orientations were designed into samples that were flat plates.
After preparing the samples, the channels were filled with water and tested in a fluid bath using through transmission ultrasonics. The results of this study showed that one could obtain sheets with different average wave transmission speeds based on the type of microstructure designed into the sample.

In this chapter the sample preparation is described first, including the different geometries of channels introduced through rapid prototyping. This is followed by a description of the ultrasonic method used to measure the response. The chapter ends with a description of the ultrasonic wave speed results obtained and a discussion of the effect of each channel topology on the measured wave speeds.

### 3.2 Materials and sample preparation

Rapid prototyping was used to construct one solid and seven structured samples from a pseudo-PC material suitable for the processing technique. The structure produced was in the form of plates with different density, size, and orientation of hollow channels. The rapid prototyping method constructs the object by depositing it layer by layer from a 3-D CAD model. The accuracy of the process allows construction of channels in sub-millimeter size.

Seven structured samples and one solid pseudo-PC (reference) sample were constructed in the form of two plates of approximately 6.25 mm thickness. Each sample occupied one quarter of the plate and was constructed with channels running parallel to the plate surface. By selecting the channel size, density and orientation we were able to construct seven different microstructures and one solid sample to be used as reference.
Figure 10 shows the eight samples. The pseudo-PC material is transparent so that the patterns shown are from the channels.

![Figure 10: Structured pseudo-polycarbonate samples.](image)

The details of the sample geometry are as follows. Sample #1 is the reference sample made with the same process but without any channels. Sample #2 consists of 24 by 24 channels crossing each other in two layers each side. The cross section of each channel is about 1.0173 mm by 1.0173 mm as shown in Figure 11.

![Figure 11: Structured pseudo-PC sample #2.](image)

Sample #3 is composed of 50 in line channels with a cross sectional dimension of each channel being about 1.0173 mm by 1.0173 mm as shown in Figure 12.
Sample #4 is a 16 by 7 crossed channels structure where each channel has a rectangular cross section of dimension about 1 mm by 5.5 mm as shown in Figure 13.

Sample #5 is composed of 23 in line rectangular cross section channels with dimensions about 1mm by 4 mm; the overlapping between the channels is about 0.8250 mm as shown in Figure 14.

Sample #6 is composed of 23 in line channels with rectangular cross sectional dimensions about 1mm by 5.5 mm; the overlapping between the channels is 2.3250 mm as shown in Figure 15.
Sample #7 is a 16 by 7 crossed channel structure with square cross sectional dimensions of about 1.5 mm by 1.5 mm as shown in Figure 16.

![Figure 16: Structured pseudo-PC sample #7.](image)

Sample #8 is composed of 23 in line channels with square cross sectional dimensions of about 1.5 mm by 1.5 mm as shown in Figure 17.

![Figure 17: PC-like structured sample #8.](image)

### 3.3 Experiments conducted

A technique used was based on sending ultrasonic waves through the samples to characterize the effects of the microstructures on the wave speeds and wave shapes. Measurements were performed using two 5 MHz immersion transducers with a focus in water of 3 inches (7.62 cm). For each samples, 600 points were scanned over an area of 12 mm x 8 mm with a resolution of about 0.4 in both the x and y directions. The focal length of the transmitter and the receiver are situated in the middle of the sample, i.e. about 3.14 mm from the edges.
Before each measurement, samples were submerged and the channels were filled with water using a syringe. The aim of this step was to allow the ultrasonic waves to pass through the sample without being attenuated by air bubbles contained in the structure of the samples. The aim of this effort was to see if we can construct a microstructure that shows a response similar to that seen in complex structured materials such as seen in human bone.

3.4 Results

Wave velocity measurements were taken using ultrasonic contact transducers and immersion transducers for the solid sample. For the seven structured samples, measurements were taken only using the immersion transducers.

For the solid sample, three different contact transducers with frequencies of 1 MHz, 7.5 MHz and 10 MHz were used. Wave velocities were determined by time of flight between front and back walls. Table 1 provides the result of the wave speed measurements.

**Table 1**: Solid sample wave speeds in function of the frequency.

<table>
<thead>
<tr>
<th>Frequency (MHz)</th>
<th>Wave Speed (m.s(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2565 ± 3</td>
</tr>
<tr>
<td>7.5</td>
<td>2584 ± 2</td>
</tr>
<tr>
<td>10</td>
<td>2590 ± 2</td>
</tr>
</tbody>
</table>
The wave speeds of the seven structured samples were determined using the through transmission method with two immersion transducers in a tank filled with water. The focus of the transducers in water is 3 inches (7.62 cm). By measuring the time of flight between the two transducers the wave speed in water can be calculated. The time $t_w$ to go through the water was then determined by dividing the water distance (which is defined by two times the focal length minus the thickness of the sample) by the wave speed in water.

The different samples were successively placed at the focal distance and the signal provided by this setup recorded. From those records the time $t$ needed for the wave to go from the transmitter to receiver (i.e. water plus sample) was measured. The time to pass through the sample $t_s$ is then given by $t_s = t - t_w$.

The average results obtained for the eight samples are given in Table 2. These are the average of measurements at 600 points on a 12 mm x 8 mm area with a resolution of about 0.4 in both the x and y directions.
Table 2: Measured sound speeds for the solid pseudo-PC and seven structured pseudo-PC samples.

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Time $t$ (μs)</th>
<th>Thickness (mm)</th>
<th>Water Distance (mm)</th>
<th>Time in Water $t_w$ (μs)</th>
<th>Time in Material $t_s$ (μs)</th>
<th>Speed of Sound (m.s$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>101.262</td>
<td>6.26</td>
<td>146.14</td>
<td>98.834</td>
<td>2.428</td>
<td>2580</td>
</tr>
<tr>
<td>2</td>
<td>101.282</td>
<td>6.29</td>
<td>146.11</td>
<td>98.815</td>
<td>2.467</td>
<td>2550</td>
</tr>
<tr>
<td>3</td>
<td>101.407</td>
<td>6.29</td>
<td>146.11</td>
<td>98.813</td>
<td>2.594</td>
<td>2427</td>
</tr>
<tr>
<td>4</td>
<td>101.723</td>
<td>6.26</td>
<td>146.14</td>
<td>98.835</td>
<td>2.888</td>
<td>2168</td>
</tr>
<tr>
<td>5</td>
<td>101.606</td>
<td>6.28</td>
<td>146.12</td>
<td>98.820</td>
<td>2.786</td>
<td>2256</td>
</tr>
<tr>
<td>6</td>
<td>101.719</td>
<td>6.18</td>
<td>146.22</td>
<td>98.892</td>
<td>2.827</td>
<td>2185</td>
</tr>
<tr>
<td>7</td>
<td>101.278</td>
<td>6.29</td>
<td>146.11</td>
<td>98.813</td>
<td>2.465</td>
<td>2553</td>
</tr>
<tr>
<td>8</td>
<td>101.301</td>
<td>6.28</td>
<td>146.12</td>
<td>98.823</td>
<td>2.478</td>
<td>2534</td>
</tr>
</tbody>
</table>

The results found in Table 2 show that the structure of the samples influences the wave speed of the material. Knowing that the wave speed measured in water is 1479 m.s$^{-1}$ and the wave speed in the solid PC is 2580 m.s$^{-1}$, the variation of the wave speed is explained when the sample has water filled channels. The amount of this change should be somewhat related to the fraction of the total cross section covered by channels. This is consistent with the fact that the lowest values of wave speeds are found for samples #6 and #4 for which the surface of holes is maximal. In contrast, in line or transverse holes of small dimensions have only a limited effect on the wave speed (i.e. the wave speed was close to the solid pseudo-PC wave speed). Figure 18 shows the wave speed as a percent of the volume occupied by water filled channels.
Figure 18: Wave speed of the structured pseudo-PC in function of the percentage of the volume occupied by channels in function of the wave.

The wave signals that were recorded were then analyzed using a fast Fourier transform (FFT) program. Plots presented below show the FFT magnitude of the signal recorded as a function of the frequency, with the solid PC results shown as a reference.

Sample #2, presented in Figure 19, has a cross hatched structure composed of a high density of channels with 50 channels with a dimension about 1.0173 by 1.0173 mm. As shown in Figure 21, the FFT magnitude wave signal of sample #2, compared to the solid pseudo-PC, is much more attenuated over the frequency domain. In addition, at around 4 MHz, 5.5 MHz and 7 MHz large drops in the FFT magnitude are observed.

Sample #3, presented in Figure 20, has the same number of channels with the same dimension as sample #2, i.e. 50 channels with a dimension about 1.0173 by 1.0173 mm. The main difference between these two samples is that sample #3 has an in-line
structure. The FFT magnitude wave signal of sample #3, compared to the solid pseudo-PC signal, is much more attenuated over the frequency domain. In contrast to sample #2, drops at 4 MHz and 7 MHz are not observed and less important at 5.5 MHz.

Figure 19: FFT magnitude Samples #2.

Figure 20: FFT magnitude sample #3.
As mentioned the main difference between these two structures is that one has an in-line structure while the other has a cross hatched structure. The influences of these two structures on the attenuation are observed Figure 21 with $\Delta \alpha(\omega)$ the difference of the attenuations between the solid pseudo-PC $\alpha_R(\omega)$ and structured pseudo-PC $\alpha_S(\omega)$ defined by

$$[\alpha_R(\omega) - \alpha_S(\omega)]d = \ln \left( \frac{|FFT(f_S(t))|}{|FFT(f_R(t))|} \right)$$

(6)

If we compare, in term of areas under the peaks, the influence of the structure of the sample #2 on the attenuation is more important than the sample #3 and in particular for some frequencies.

![Figure 21](image)

**Figure 21:** Difference of the attenuations between the solid PC and structured PC for samples #2 and #3.

Results for sample #4 are presented Figure 22. Composed of 23 channels; this sample is considered as having a low density of channels. Sample #4 has a cross hatched
structure with channel cross sections about 1 mm by 5.5 mm. The FFT magnitude for this sample shows a relatively low attenuation of the signal.

Results for sample #5 are presented Figure 23. Sample #5 has an in-line structure with an overlap of 0.8250 mm and is composed of 23 channels with a dimension about 1.0 mm by 4.0 mm. Compared to the FFT magnitude signal of sample #4, the signal of sample #5 is similar up to 4 MHz and between 4 MHz and 6 MHz and after 7 MHz the attenuation is much larger.

Sample #6, presented in Figure 24, has an in-line structure and is composed of 23 channels with a dimension about 1.0 mm by 5.5 mm with an overlap between the channels of 2.3250 mm. Its FFT magnitude wave signal is similar to sample #4, i.e. the FFT magnitude for this sample shows a relatively low attenuation of the signal.

![FFT Magnitude Sample #4](image)

**Figure 22**: FFT magnitude sample #4.
The difference of the attenuations $\Delta a(\omega)$ between the solid pseudo-PC and structured pseudo-PC for samples #4, #5 and, #6 over the frequency domain is shown Figure 25. In addition to the variation of overlap and dimension of the channels, two
structures are presented, an in line and a cross hatched. If we compare, in term of areas under the peaks, the influence of the structures of the samples #4 and #6 is minor. In contrast, over 4 MHz the influence of the structure of sample #5 is noticeable.

**Figure 25**: Difference of the attenuations between the solid pseudo-PC and structured pseudo-PC for samples #4, #5, and #6.

Plot of the FFT magnitude versus frequency for sample #7 is presented Figure 26. Sample #7 has a cross hatched structure composed of 23 channels with a dimension about 1.5 mm by 1.5 mm. The attenuation over the frequency domain is relatively constant with some slight variations of the signal.

Sample #8, presented Figure 27, has an in-line structure composed of 23 holes with a dimension about 1.5 mm by 1.5 mm. For this sample compared to the reference sample the attenuation of the FFT magnitude signal is very significant to certain points in particular around 3 MHz, 5 MHz and 7 MHz.
As mentioned, the main difference between these two structures is that one has an in-line structure while the other has a cross hatched structure. The influences of these two structures on the attenuation are observed Figure 28 with $\Delta a(\omega)$ the difference of the
attenuations between the solid pseudo-PC and structured pseudo-PC. If we compare, in term of areas under the peaks, the influence of the structure of sample #8 on the attenuation is more important than sample #7 but in both cases for the same frequencies drops are observed.

![Figure 28: Difference of the attenuations between the solid pseudo-PC and structured pseudo-PC for samples #7 and #8.](image)

**Figure 28:** Difference of the attenuations between the solid pseudo-PC and structured pseudo-PC for samples #7 and #8.

### 3.5 Conclusion

As has been shown, by designing microstructures into solid sheets we can change the acoustic wave speed, wave form and attenuation. In this example, we used a rapid prototyping system that used a pseudo-polycarbonate material to construct sheets with different number, size and orientation of cylindrical channels. We filled the channels with water and studied the resulting systems. The results show that we can, on average, vary the wave speed between that of solid pseudo-PC material and that for water. In addition, it was shown that different patterns produce different wave signal transformations. This
was observed through a study of the FFT of the measured signals. This study indicates that we can use different microstructures to obtain desired characteristics in a structured material, and by selecting the solid and fluid phases influence the overall properties.

For samples with a low percent of volume occupied by channels the consequence is that the influence on the wave speed is negligible and this does not depend on whether or not the channels have in-line or cross-hatched structure. In contrast, the lowest wave speeds resulted from samples with a high percent of the volume occupied by channels. The organization of the structure in-line or cross-hatched, does influence to some extent the wave speed. It appears that in order to control the material wave speed response the percentage of volume occupied by the channels is the parameter that has the largest influence. Obviously the higher the percent of volume occupied by channels, the lower the wave speed.

The influence of the organization of the structure and the percentage of volume occupied by channels is more difficult to relate to the attenuation. It appears that, in terms of areas under the peaks, samples #4 and #6 which have a percentage of volume occupied by channels about 40% are less attenuated. In contrast, samples with a low percentage of volume occupied by channels like samples #2, #3 and #8 are more attenuated. If we compare these two sets of samples it seems that the influence of the orientation of the channels, i.e. in-line or transverse, is negligible. The attenuation is relatively low for samples #4, #5, and #6, which have a high percent of volume occupied by channels (both in-line and cross-hatched structures).
In contrast to the wave speed the structure affect on attenuation is not clearly define. Even if similarities in the material response as a function of structure appear these changes are not significant enough, or repeatable enough, to suggest one cause to the modification of the attenuation. More tests with different void channel structures should be conducted to define more precisely how the structures influence the response.
Chapter 4

ULTRASONIC INSPECTION ON BONES

4.1 Introduction

Bone structure is considered complex because it is composed of both a compact structure and a porous structure making it difficult to characterize the material properties. Through the decades numerous studies have been conducted to develop models [15][16] and ultrasonic experimental methods to measure and define at low-megahertz frequencies the material properties [17][18]. An ultrasonic setup was used to characterize this material and to use the results to improve the structured PC.

Bovine bone samples were acquired and inspected by ultrasound. The purpose of this study was to characterize the wave speed and attenuation in bone using contact and immersion transducers.

This chapter is organized as follow. First a description of bones is given and followed by a description of the bone samples and their preparation. Then, the different
experimental methods conducted on bones are presented. The chapter ends with the ultrasonic results.

4.2 Introduction to bone

Bone tissue is composed of organic and inorganic phases. The mineral phase, which represents 70% of the tissue by weight, is mostly composed of crystals of calcium and phosphate. The other 30% corresponds to the organic or extracellular matrix. According to their shapes, four kinds of bones can be defined: short bones (wrist, ankle), flat bones (ribs, sternum), long bones (humerus, femur) and irregular bones (hip bones, vertebrae) [2].

In addition of their shapes, bone tissues can be identified based on function of the osseous tissues as: trabecular bone (cancellous or spongy bone) and cortical bone (compact bone). Cortical bone has four times the mass of trabecular bone. Such a mass difference can be explained by the high porosity of trabecular bone ranging from 50% to 90%. The cortical bone represents 80% of the bone mass and gives bones their smooth, white and solid appearance, and protection of the internal part of the bone. The internal part or trabecular bone represents 20% of the total bone mass [16][19]. Figure 29 shows the organization of the bone structure.
Figure 29: Bone structure composed of the cortical bone which surrounds the trabecular bone [20].

4.3 Material used and sample preparation

For the experiments conducted, cow femur bones have been used. These bones, shown in Figure 30, are longer than wide and are categorized as long bones and are mostly composed of compact bone. The sample length varies from 8 cm to 15 cm.

Figure 30: Cow femur bones.
In order to get flat and smooth samples, the samples were machined. Using a cutting machine and a polishing machine samples with a thickness varying from 3 mm to 5 mm were prepared, as shown on Figure 31.

![Figure 31: Bone samples used during the experiments.](image1)

When the samples were cut out, they were placed in a frame specially designed to hold the samples during the experiments, shown Figure 32.

![Figure 32: Immersion tank and fixtures.](image2)
4.4 Experiments conducted

The primary purpose of this study was to determine the properties of bovine bone samples. In support of this study ultrasonic tests were performed using both contact transducers and immersion transducers.

Pulse echo technique using contact transducers of 7.5 MHz and 10 MHz were used to determine, as a first approximation, the wave speed, the wave modulus and the attenuation of the cow bone samples. The low porosity of the samples was why contact measurements were successfully realized using reflected wave signals.

The tests needing the use of immersion transducers were done in an immersion tank. The frequency inspected was 1 MHz using an unfocused immersion transducer and a focus immersion transducer with a focus length of 4 inches. To ensure that the transducer receives a uniform wave signal with maximum amplitude, the distance N, known as the near field distance, was calculated. In fact, because of wave interference near the transducer, fluctuations in the sound intensity may lead to inaccuracy in flaw detection. Beyond the near field, the signal is more uniform but also decreases with the distance, this is why the distance N, which is the transition between those two areas, is chosen as the distance at which the transducer is positioned from the sample. A schematic representation of this phenomenon is given Figure 33.
The near field distance $N$ is calculated using the equation

$$N = \frac{D^2}{4\lambda} \left[ 1 - \left( \frac{\lambda}{D} \right)^2 \right],$$

where $\lambda$ is the wavelength and $D$ the diameter of the transducer. The unfocused immersion transducer used during the experiments had a diameter about 1 inch and a frequency of 1 MHz, and according to the equation (7) the near field distance was 4.235 inches. As a result, this transducer was positioned 4.235 inches from the middle of the bone sample.

The transmission method was used to perform the experiments. For highly attenuating materials, such as cancellous bone, the transmission mode is more practical since the acoustic wave passes through the material only once so the measured signal is less attenuated [2].

Experiments were performed with inclined sonification. Longitudinal and shear wave modes were calculated using the first critical angle determined by the Snell’s law.
using the contact measurements. Schematic representation of the transmission method in the water bath is found Figure 34. For the tests performed, only the transmitting transducer moves while the receiver stays perpendicular to the bone sample.

![Diagram of through transmission method](image)

**Figure 34:** Schematic representation of the through transmission method.

The parameters used for these experiments are found in Table 3.

**Table 3:** Through transmission experimental settings.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Bone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (MHz)</td>
<td>1</td>
</tr>
<tr>
<td>Gain (dB)</td>
<td>20</td>
</tr>
<tr>
<td>Pulse Amplitude</td>
<td>4</td>
</tr>
<tr>
<td>Pulse Energy</td>
<td>1 Low</td>
</tr>
<tr>
<td>Damping</td>
<td>1</td>
</tr>
</tbody>
</table>
4.6 Results

Wave velocity measurements were taken using longitudinal ultrasonic contact transducers for the cow bone sample. To perform these tests two different contact transducers with frequencies about 7.5 MHz and 10 MHz were used. Wave velocities were determined by time of flight between front and back walls.

As illustrated Figure 35, the first critical angle for the bone sample tested was defined theoretically using the Snell's law

$$\theta_c = \sin^{-1} \left( \frac{c_1}{c_2} \right)$$  \hspace{1cm} (8)

where $\theta_c$ is the first critical angle and $\theta_2$ the refracted angle equal to 90°, and $c_1$ is the wave velocity in material 1 and $c_2$ is the wave velocity in material 2. In this case, all of the longitudinal energy is either reflected or converted to an interface wave. Only shear waves remain in the second material.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{snell.png}
\caption{Diagram of the Snell’s law.}
\end{figure}
Attenuation was calculated for the material at 7.5 MHz and 10 MHz using contact transducers. The equation is given by

\[ e^{-2\alpha d} = \frac{|F_2(\omega)|}{|F_1(\omega)|}. \]  

(9)

the quantity \( \alpha \) is the attenuation coefficient of the wave and is expressed in Nepers per length, and \( d \) is the sample thickness. \( F_1(\omega) \) and \( F_2(\omega) \) are the Fourier transforms of two successive reflections.

For the samples tested, the wave speeds, the attenuations and the critical angles are provided Table 4.

**Table 4:** Ultrasonic inspection results on bone sample as a function of the frequency using contact transducers.

<table>
<thead>
<tr>
<th>Frequency (MHz)</th>
<th>Attenuation (Np.m(^{-1}))</th>
<th>Wave Speed (m.s(^{-1}))</th>
<th>Critical Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.5</td>
<td>500</td>
<td>3228</td>
<td>27°</td>
</tr>
<tr>
<td>10</td>
<td>680</td>
<td>3251</td>
<td>27°</td>
</tr>
</tbody>
</table>

Considering the error of the measurements and the fact that two 1 MHz immersion transducers are used to perform the tests in the tank, the first critical angle for the bone sample is considered to be comprised between 25° and 30°.

Through transmission tests were performed in a tank filled with water and two 1 MHz immersion transducers fixed on a turntable. Through transmission signal, presented Figure 36, was recorded with and without a bone sample in order to characterize the
signal loss when the wave goes through the sample. As expected the FFT magnitude of 
the bone signal is lower than the FFT magnitude of the water signal.

**Figure 36**: Bone and water through transmission signals using two 1 MHz immersion 
transducers.

Several sets of measurements were performed both for the longitudinal wave 
speed and for the wave speed using a rotation corresponding to the first critical angle but 
the results were off and not repeatable. Wave speeds results varied from 1500 m.s\(^{-1}\) to 
3200 m.s\(^{-1}\).

4.7 Conclusion

During this study bovine bone samples were analyzed using ultrasonic methods to 
characterize the material properties such as the wave speed and the attenuation. The 
inhomogeneous porous structure of bone samples makes difficult the determination of 
these parameters.
Contact transducers were used as a first approximation to determine the first critical angle and gave coherent results since in accordance with the work conducted by [22]. Wave speeds measurements were also coherent with the values found in the literature and notably with [2], in which values of wave speeds in cortical bones are measured between 3000 m.s$^{-1}$ and 4000 m.s$^{-1}$.

In contrast with the tests performed with contact transducers, through transmission results were off. Numerous reasons can explain the large error in the results. These may be that the transducers are not well aligned or the distance between the two transducers is not precise. The influence of these on the results can be large. In addition, a better ultrasonic setup and measurements procedure may help. Other kinds of tests, like polar transmission to observe the scattering, may provide the effect of the microstructure.
Chapter 5

QUANTIFY VISCOELASTIC PROPERTIES OF SOFT TISSUES

5.1 Introduction

The attenuation, particularly in shear, for soft tissues is so large that methods that require the wave to travel through the sample normally cannot be used. Simply, the wave attenuates so fast that one cannot obtain a strong enough signal, particularly if multiple echoes are needed for the measurement. It has been demonstrated that in the low frequency domain for soft tissues the sound speeds are of the order of 10-100 m.s\(^{-1}\) and the shear wave attenuation coefficients are of the order of 10000 times those of longitudinal waves [23][24]. This strong attenuation is also present at megahertz frequencies in soft tissues; this is why transmission techniques are not practical for measuring the shear properties. In such materials, a method that can extract the response without following the wave though the tissue can be very useful. One such method uses the characteristics of the reflected wave from the surface of a sample to obtain the desired
wave speed and attenuation in the sample. This is the ultrasonic impedance method and was developed by Mason and co-workers in 1949 [25]. In this chapter we study this method and evaluate the possibility to measure wave speed and attenuation for two test materials.

The two materials selected for this study are poly (methyl methacrylate) (PMMA) and polydimethylsiloxane (PDMS). PMMA is a well studied glassy polymer and PDMS is a soft rubbery polymer used in prostheses made to replace soft tissue like skin.

The results of this study are organized in this chapter by first describing the method and giving the theoretical base of the method. This is followed by some typical results, that are used to show how information from the signal can be extracted and the expected accuracy. Then the characteristics of PMMA and PDMS are described. This is followed by a description of the results of the proposed method for PMMA and PDMS and a comparison to expected results.

5.2 Theoretical background on wave propagation

Measurements of the incident and reflected wave from an interface between two materials can be used to characterize the properties of the material beyond the interface. In this section we use one-dimensional time harmonic attenuating plane shear waves to show how this method works for linear viscoelastic media.
Figure 37: Reflection at boundary between two medium with normal incidence.

Let us first consider the interface problem for which an incident wave partially reflects from the interface and partially transmits through it. Figure 37 shows a typical interface between media 1 and 2. The general solution to this problem in terms of the space variable $x$ along the direction of the wave and the time variable $t$ is characterized by stress field $\sigma(x,t)$ and a displacement field $u(x,t)$ which are appropriately connected through the constitutive character of the material. Since the behavior of the material in each medium is different, we write the balance of momentum for each medium and enforce continuity conditions of stress and displacement at the interface to connect the solution to each side. Let $x < 0$ denote media 1 and $x > 0$ denote media 2. Balance of momentum equations for the two media are given by

$$\begin{align*}
\nabla \cdot \sigma + \rho \cdot b &= \rho \cdot a \quad \text{for } x < 0, \\
\nabla \cdot \sigma + \rho \cdot b &= \rho \cdot a \quad \text{for } x > 0,
\end{align*}$$

(10)

where $\rho$ is the density, $b$ is the body force and $a = \ddot{u}$ is the acceleration. The continuity of the displacement and of the normal traction are written
\[
\begin{aligned}
\begin{cases}
  u(-0, t) = u(+0, t), \\
  \sigma(-0, t)n = \sigma(+0, t)n,
\end{cases}
\end{aligned}
\]  
(11)

where \(n\) is the normal to the surface.

We will now solve these equations for a shear wave and ignore the body force. The only non-trivial balance equations become

\[
\begin{aligned}
\begin{cases}
  \frac{\partial \sigma}{\partial x} = \rho \frac{\partial^2 u}{\partial t^2} & \text{for } x < 0, \\
  \frac{\partial \sigma}{\partial x} = \rho \frac{\partial^2 u}{\partial t^2} & \text{for } x > 0,
\end{cases}
\end{aligned}
\]

where \(\sigma(x,t)\) is the shear stress and \(u(x,t)\) is the transverse displacement. The only non-trivial conditions of continuity for the shear wave become

\[
\begin{aligned}
\begin{cases}
  u(-0, t) = u(+0, t), \\
  \sigma(-0, t) = \sigma(+0, t).
\end{cases}
\end{aligned}
\]  
(13)

We can separate the displacement in medium 1 into that of the incident wave \(u_i(x,t)\) and the reflected wave \(u_r(x,t)\), and denote the transmitted wave into medium 2 as \(u_t(x,t)\). We can then rewrite the continuity equation for the displacement as

\[
u_i(-0,t) + u_r(-0,t) = u_t(+0,t).
\]  
(14)

### 5.3 Attenuating harmonic waves in a viscoelastic materials

We will first consider the problem of an attenuating harmonic shear wave traveling in a single medium and solve for the response and then connect the two media through the
continuity equations in the following section. For a single medium we have the balance law

\[
\frac{\partial \sigma(x,t)}{\partial x} = \rho \frac{\partial^2 u(x,t)}{\partial t^2}. \tag{15}
\]

We assume harmonically attenuating plane shear waves given by an equation of the form

\[
u(x,t) = u_0 \exp(-\alpha x) \cos[\omega(t - \frac{x}{c})], \tag{16}
\]

where \(u_0\) is the amplitude, \(\alpha\) is the attenuation coefficient, \(\omega\) is the circular frequency, and \(c\) is the shear wave speed. As seen in Figure 38, the wave is sinusoidal with constant amplitude at each location in space, but attenuates with increasing \(x\). This results in an acceleration described by

\[
\frac{\partial^2 u(x,t)}{\partial t^2} = -\exp(-\alpha x) \omega^2 \cos[\omega(t - \frac{x}{c})]. \tag{17}
\]

\[\text{Figure 38: Infinitesimal shear sinusoidal attenuating waves in time.}\]

The material is assumed to be a viscoelastic material defined by a constitutive equation of the form
\[ \sigma(t) = G(0)\gamma(t) + \int_0^t G'(t-\tau)\gamma(\tau)d\tau, \tag{18} \]

where \( G(t) \) is the relaxation function having a form like that given in Figure 39, \( G'(t) \) is its derivative, and \( \gamma(t) \) is the shear strain given in this problem as

\[ \gamma = \frac{\partial u}{\partial x}. \tag{19} \]

The shear strain can be calculated from the shear displacement by taking the derivative with respect to position, given by

\[ \gamma(t) = \frac{\partial u(x,t)}{\partial x} = u_0 \exp(-\alpha x) \left\{ -\alpha \cos(\omega(t - \frac{x}{c})) + \frac{\omega}{c} \sin(\omega(t - \frac{x}{c})) \right\}. \tag{20} \]

We can substitute this back into the constitutive equation to get the shear stress associated with this harmonically attenuating wave.

We next take its derivative to obtain
\[
\frac{\partial \sigma(x,t)}{\partial x} = u_0 \exp(-\alpha x)[G(0)(\alpha^2 - \frac{\omega^2}{c^2}) \cos[\omega(t - \frac{x}{c})] - \frac{2\alpha \omega}{c} G(0) \sin[\omega(t - \frac{x}{c})]]
+ (\alpha^2 - \frac{\omega^2}{c^2}) \int_0^\infty G'(t - \tau) \cos[\omega(\tau - \frac{x}{c})] d\tau - \frac{2\alpha \omega}{c} \int_0^\infty G'(t - \tau) \sin[\omega(\tau - \frac{x}{c})] d\tau].
\]

Once we substitute this and the equation for the acceleration into the balance law, we obtain the equation

\[
[G(0)(\alpha^2 - \frac{\omega^2}{c^2} + \rho \omega^2) \cos[\omega(t - \frac{x}{c})] + [-2G(0) \frac{\alpha \omega}{c} \sin[\omega(t - \frac{x}{c})]]
+ (\alpha^2 - \frac{\omega^2}{c^2}) \int_0^\infty G'(s) \cos[\omega(t - s - \frac{x}{c})] ds + (-2\frac{\alpha \omega}{c}) \int_0^\infty G'(s) \sin[\omega(t - s - \frac{x}{c})] ds = 0,
\]

which must be satisfied for each point \(x\).

For times that are large relative to the relaxation time, the integrals approach constant values and one can separate this equation into \(\sin(\omega t)\) and \(\cos(\omega t)\) terms. Setting the coefficient of each equal to zero results in two equations that can be written as

\[
\int_0^\infty G'(s) \sin(\omega s) ds = \frac{-2\rho c^3(\omega)\alpha(\omega)\omega^3}{[c^2(\omega)\alpha^2(\omega) + \omega^2]^3}, \quad (23)
\]

\[
\int_0^\infty G'(s) \cos(\omega s) ds = -G(0) + \frac{-\rho c^2(\omega)\omega^2 [c^2(\omega)\alpha^2(\omega) - \omega^2]}{[c^2(\omega)\alpha^2(\omega) + \omega^2]^2}, \quad (24)
\]

where the wave speed \(c(\omega)\) and attenuation \(\alpha(\omega)\) are functions of frequency \(\omega\). These can be inverted to give the wave speed and attenuation coefficients in terms of the relaxation function.
5.4 Reflection and transmission of waves at interfaces

For the application that we are considering, we have two materials in contact with each other as shown in Figure 40. The silica plate is our reference material and the soft tissue is the material that we are trying to characterize in terms of its wave speed and attenuation for the given incident wave frequency. We will denote the silica as material \( a \) and the soft tissue as material \( b \). A plane sinusoidal wave travels along the \( y \)-direction and partially gets reflected from the interface and partially gets transmitted into the soft tissue.

**Figure 40**: Schematic representation of the experimental setup.

For this application, we write the incident, reflected and transmitted waves as

\[
\begin{align*}
    u_i(y,t) &= e^{-\alpha_i y} \cos \left[ \omega(t - \frac{y}{c_i}) \right], \quad (25) \\
    u_r(y,t) &= A_i e^{-\alpha_i(y-d)} \cos \left[ \omega(t - \frac{d-y}{c_i}) + \phi_i \right], \quad (26) \\
    u_t(y,t) &= A_t e^{-\alpha_t(y-d)} \cos \left[ \omega(t - \frac{y-d}{c_t}) + \phi_t \right], \quad (27)
\end{align*}
\]
where we have rotated and move the coordinate so that \( y = d \) is the interface, and where \( i \) denotes incident, \( t \) denotes the transmitted, and \( r \) denotes reflected, and the incident and reflected wave speed and attenuation coefficient are taken to be the same.

Interface displacement continuity at \( y = d \) is written as

\[ u_i(d,t) + u_r(d,t) = u_r(d,t). \tag{28} \]

After substitution of the wave equations into the displacement continuity requirement, we obtain one equation from the coefficient of the sine and one from the coefficient of the cosine term. These are

\[ e^{-\alpha_i d} \cos\left(\frac{\alpha d}{c_i}\right) + A_i \cos(\phi_i) = A_i \cos(\phi_i), \tag{29} \]

\[ e^{-\alpha_i d} \sin\left(\frac{\alpha d}{c_i}\right) + A_i \sin(\phi_i) = A_i \sin(\phi_i). \tag{30} \]

To write the stress continuity, we need the total shear strains in media 1 and 2. In media 1 we have both the incident and reflected waves, and in 2 we have the transmitted waves. From the displacements we obtain the following strains

\[ \gamma_i(y,t) + \gamma_r(y,t) = \alpha_i e^{-\alpha_i y} \cos[\omega(t - \frac{y}{c_i})] + \frac{\omega}{c_i} e^{-\alpha_i y} \sin[\omega(t - \frac{y}{c_i})] \]

\[ + A_r e^{-\alpha_i(d-y)} \alpha_i \cos[\omega(t - \frac{d-y}{c_i}) + \phi_r] \]

\[ - A_r \frac{\omega}{c_i} e^{-\alpha_i(d-y)} \sin[\omega(t - \frac{d-y}{c_i}) + \phi_r], \tag{31} \]
\[ \gamma_i(y,t) = A_i e^{-\alpha_i(y-d)} \left\{ -\alpha_i \cos(\omega(t - \frac{y-d}{c_i}) + \phi_i) + \frac{\omega}{c_i} \sin(\omega(t - \frac{y-d}{c_i}) + \phi_i) \right\}. \] (32)

If we substitute this into the expression for stress we get the stress fields in media \(a\) and \(b\) as:

\[ \sigma_a = G_i(0) \left\{ -\alpha_i e^{-\alpha_i y} \cos(\omega(t - \frac{y}{c_i}))+ \alpha_i e^{-\alpha_i y} \frac{\omega}{c_i} \sin(\omega(t - \frac{y}{c_i}))+ A_i e^{-\alpha_i(y-d)} \alpha_i \cos(\omega(t - \frac{d-y}{c_i}) + \phi_i) - A_i e^{-\alpha_i(y-d)} \frac{\omega}{c_i} \sin(\omega(t - \frac{d-y}{c_i}) + \phi_i) \right\} \] (33)

\[ = G_i(0) \left\{ -\alpha_i e^{-\alpha_i y} \cos(\omega(t - \frac{y}{c_i}))+ \alpha_i e^{-\alpha_i y} \frac{\omega}{c_i} \sin(\omega(t - \frac{y}{c_i}))+ A_i e^{-\alpha_i(y-d)} \alpha_i \cos(\omega(t - \frac{d-y}{c_i}) + \phi_i) - A_i e^{-\alpha_i(y-d)} \frac{\omega}{c_i} \sin(\omega(t - \frac{d-y}{c_i}) + \phi_i) \right\} \] (34)

At the interface we set these two stresses equal and obtain one equation from the coefficient of the sine term and one from the coefficient of the cosine term. These are

\[ A_i \sqrt{(-\alpha_i)^2 + \left( \frac{\omega}{c_i} \right)^2} \left[ h_i(\omega) \cos(\phi_i + \eta) + f_i(\omega) \sin(\phi_i + \eta) \right] \]

\[ = \sqrt{(-\alpha_i)^2 + \left( \frac{\omega}{c_i} \right)^2} \left[ h_i(\omega) \left[ e^{-\alpha_i d} \cos(\theta - \frac{\omega d}{c_i}) - A_i \cos(\phi_i + \theta) \right] + f_i(\omega) \left[ e^{-\alpha_i d} \sin(\theta - \frac{\omega d}{c_i}) - A_i \sin(\phi_i + \theta) \right] \right] \] (35)

\[ A_i \sqrt{(-\alpha_i)^2 + \left( \frac{\omega}{c_i} \right)^2} \left[ h_i(\omega) \sin(\phi_i + \eta) + f_i(\omega) \cos(\phi_i + \eta) \right] \]

\[ = \sqrt{(-\alpha_i)^2 + \left( \frac{\omega}{c_i} \right)^2} \left[ h_i(\omega) \left[ e^{-\alpha_i d} \sin(\theta - \frac{\omega d}{c_i}) - A_i \sin(\phi_i + \theta) \right] + f_i(\omega) \left[ e^{-\alpha_i d} \cos(\theta - \frac{\omega d}{c_i}) - A_i \cos(\phi_i + \theta) \right] \right] \] (36)
where,

\[ f_i(\omega) = \int_{0}^{\infty} G_i'(s) \sin(\omega s) ds, \quad (37) \]

\[ h_i(\omega) = G_i(0) + \int_{0}^{\infty} G_i'(s) \cos(\omega s) ds, \quad (38) \]

\[ f_i(\omega) = \int_{0}^{\infty} G_i'(s) \sin(\omega s) ds = \frac{-2\rho_i c_i^3 \omega^3}{(c_i^2 - \alpha_i^2 + \omega^2)^2}, \quad (39) \]

\[ h_i(\omega) = G_i(0) + \int_{0}^{\infty} G_i'(s) \cos(\omega s) ds = \frac{-\rho_i c_i^2 \omega^2 (c_i^2 - \alpha_i^2 - \omega^2)}{(c_i^2 - \alpha_i^2 + \omega^2)^2}, \quad (40) \]

and,

\[ \cos(\theta) = \frac{\omega}{c_i \sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2}}, \quad (41) \]

\[ \sin(\theta) = \frac{-\alpha_i}{\sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2}}, \quad (42) \]

\[ \cos(\eta) = \frac{\omega}{c_i \sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2}}, \quad (43) \]

\[ \sin(\eta) = \frac{-\alpha_i}{\sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2}}, \quad (44) \]
Let \( x_1 = -\alpha_i h_i(\omega) - \frac{\omega}{c_i} f_i(\omega) \) and \( x_2 = -\alpha_i f_i(\omega) - \frac{\omega}{c_i} h_i(\omega) \), after rearranging the four equations from the continuity (two for displacement and two for stress) we get:

\[
e^{-\alpha_i t} \cos\left(\frac{\omega t}{c_i}\right) + A_\phi \cos(\phi_i) = A_i \cos(\phi_i), \quad (45)
\]

\[
e^{-\alpha_i t} \sin\left(\frac{\omega t}{c_i}\right) - A_\phi \sin(\phi_i) = -A_i \sin(\phi_i), \quad (46)
\]

\[
A_i \cos(\phi_i) x_2 - A_i \sin(\phi_i) x_1 = \sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2} \left[ h_i(\omega)[e^{-\alpha_i t} \cos(\theta - \frac{\omega t}{c_i}) - A_\phi \cos(\phi_i + \theta)] + f_i(\omega)[e^{-\alpha_i t} \sin(\theta - \frac{\omega t}{c_i}) - A_\phi \sin(\phi_i + \theta)]\right], \quad (47)
\]

\[
A_i \sin(\phi_i) x_2 + A_i \cos(\phi_i) x_1 = \sqrt{(-\alpha_i)^2 + \left(\frac{\omega}{c_i}\right)^2} \left[ h_i(\omega)[e^{-\alpha_i t} \sin(\theta - \frac{\omega t}{c_i}) - A_\phi \sin(\phi_i + \theta)] + f_i(\omega)[-e^{-\alpha_i t} \cos(\theta - \frac{\omega t}{c_i}) + A_\phi \cos(\phi_i + \theta)]\right]. \quad (48)
\]

Solving these four equations results in

\[
x_1 = \frac{-2 \rho_i \omega^3 c_i A_i e^{-\alpha_i t} \sin(\phi_i) + \frac{\omega t}{c_i}}{\{e^{-2\alpha_i t} + A_i^2 + 2A_i e^{-\alpha_i t} \cos(\frac{\omega t}{c_i} + \phi_i)\}(c_i^2 \alpha_i^2 + \omega^2)}, \quad (49)
\]

\[
x_2 = \frac{-\rho_i \omega^3 c_i (A_i^2 - e^{-2\alpha_i t}) + 2A_i e^{-\alpha_i t} \rho_i \omega^2 c_i^2 \alpha_i \sin(\frac{\omega t}{c_i} + \phi_i)}{\{e^{-2\alpha_i t} + A_i^2 + 2A_i e^{-\alpha_i t} \cos(\frac{\omega t}{c_i} + \phi_i)\}(c_i^2 \alpha_i^2 + \omega^2)}. \quad (50)
\]

Upon back substitution these results give
\[
\alpha_i = \frac{\omega^2 \rho_i x_i}{x_i^2 + x_i^2}, \\
c_i = \frac{x_i^2 + x_i^2}{\omega \rho_i x_i}.
\]

5.4 Application to experimental setup

We have now developed the needed equations and will look at how we can use them to evaluate the needed material parameters. We will do it in two steps. We first consider the setup without a sample to evaluate the silica properties and then consider the silica and soft tissue together. We also consider that normal waves are a composition of different wave frequencies so we use fast Fourier transforms (FFTs) to decompose the wave into its components and study the individual responses.

If there is no medium 2, shear waves cannot be transmitted into air. This setup allows us to characterize medium 1 which is the silica. Since the shear stress is zero in the air, from the shear stress continuity condition at the interface we find

\[
(c_i^2 \alpha_i^2 - \omega^2) \sigma_r \sin(\phi_r + \theta) - 2c_i \alpha_i \omega A \cos(\phi_r + \theta) \\
= (c_i^2 \alpha_i^2 - \omega^2) e^{-\alpha_d} \sin(\theta + \frac{\alpha d}{c_i}) - 2c_i \alpha_i \omega e^{-\alpha_d} \cos(\theta + \frac{\alpha d}{c_i}),
\]

\[
(c_i^2 \alpha_i^2 - \omega^2) \sigma_r \sin(\phi_r + \theta) - 2c_i \alpha_i \omega A \cos(\phi_r + \theta) \\
= (c_i^2 \alpha_i^2 - \omega^2) e^{-\alpha_d} \sin(\theta + \frac{\alpha d}{c_i}) - 2c_i \alpha_i \omega e^{-\alpha_d} \cos(\theta + \frac{\alpha d}{c_i}).
\]
We solve these we get the amplitude and phase shift of the reflected wave in terms of the width of the silica, the wave speed, the attenuation coefficient and the frequency of the signal. These relations are

\[ A_r' = e^{-\alpha d} \], \hspace{1cm} (55)

\[ \phi_r' = -\frac{\alpha d}{c_i}. \] \hspace{1cm} (56)

Since the transducer is at the origin of the coordinate (y=0), not at the interface, we can use these parameters in the harmonic wave equation to get the displacement of the reflected wave at the origin. We do this for two different silica thicknesses and obtain

\[ u_r'(t) = e^{-2\alpha d_1} \cos(\omega_0 t - \frac{2\omega_0 d_1}{c_i}), \] \hspace{1cm} (57)

\[ u_r''(t) = e^{-2\alpha d_2} \cos(\omega_0 t - \frac{2\omega_0 d_2}{c_i}). \] \hspace{1cm} (58)

where \(\omega_0\) is the center frequency of the transducer, \(u_r'\) and \(u_r''\) are reflected waves resulting, respectively, from the different thicknesses \(d_1\) and \(d_2\). We now integrate them over one quarter of the period of the cosine wave to get

\[ I[u_r'] = \int_{T}^{T+\frac{\pi}{2\omega_0}} e^{-2\alpha d_1} \cos(\omega_0 t - \frac{2\omega_0 d_1}{c_i})dt = e^{-2\alpha d_1}, \] \hspace{1cm} (59)

\[ I[u_r''] = \int_{T}^{T+\frac{\pi}{2\omega_0}} e^{-2\alpha d_2} \cos(\omega_0 t - \frac{2\omega_0 d_2}{c_i})dt = e^{-2\alpha d_2}. \] \hspace{1cm} (60)
where $T$ is any time that a wave starts from zero. We now can solve for the attenuation using the equation

$$\alpha_i(\omega_0) = \frac{\ln(I[u_r'])}{2(d_1 - d_2)}.$$  \hspace{1cm} (61)

The wave speed can be calculated from the time the signals become zero. This relation is given by

$$c_i = \frac{2(d_1 - d_2)}{t_1 - t_2 - \frac{n\pi}{\omega_0}},$$ \hspace{1cm} (62)

where $t_1$ and $t_2$ are the times the two signals cross zero and $n$ is an integer.

For the case of silica in contact with a soft tissue, we can do a similar process. Using the same thickness silica, we get the signal once with $u_r$ and once without the tissue $u_r'$. In this case the reflected wave received by the transducer (at $y=0$) is

$$u_i(t) = A_i e^{-\alpha_i d} \cos[\omega_0 t - \frac{\omega_0 d}{c_i} + \phi_i],$$ \hspace{1cm} (63)

$$u_i'(t) = A_i' e^{-\alpha_i d} \cos[\omega_0 t - \frac{\omega_0 d}{c_i} + \phi_i'],$$ \hspace{1cm} (64)

where the prime refers to the case of silica without a tissue. We have already shown that $A_i' = e^{-\alpha_i d}$. We can integrate the two signals over one quarter of the period and then divide them to get
\[ A_r = \frac{I[u_r(t)]}{I[u_r(t)]} e^{-\alpha d}. \]  

(65)

We can find the intercept of each function with the axis and as a result obtain the phase shift from

\[ \omega_d t_0 - \frac{\omega_0 d}{c_i} + \phi_r = \omega_0 t_0 - \frac{\omega_0 d}{c_i} + \phi'_r + n\pi, \]  

(66)

which gives

\[ \phi_r = \omega_0 (t_0' - t_0) + \phi'_r + n\pi. \]  

(67)

where we know that \( \phi'_r = -\frac{\alpha d}{c_i}. \)

Once we have \( A_r \) and \( \phi_r \), we can substitute them into equations (49) and (50) to get \( x_1 \) and \( x_2 \) to then substitute into (51) and (52) to get the wave speed and attenuation of the tissue.

5.5 Description of the experimental setup

The objectives of these tests are to determine the longitudinal and shear properties of soft tissues such as brain (gel), skin (PDMS) using ultrasound. Due to the high attenuation of shear waves at megahertz frequencies in soft tissues, transmission techniques are not practical in measuring the shear properties. A reflection technique has been developed by Mason and co-workers in 1949 [25]. The basic idea behind this technique is to obtain the
shear properties through the mechanical impedance by measuring the reflection at the surface of a sample. This idea is presented Figure 41.

Figure 41: Schematic representation of the experimental apparatus.

This is one of the only ultrasonic technique which allows the determination of shear properties.

Through the decades different experimental setups based on ultrasonic transmission method have been developed. As an attempt to study the shear properties of soft tissues an ultrasonic transmission based method using a longitudinal transducer and the mode conversion block to generate shear waves was developed. Results on shear properties on tissue-like materials were reported by Madsen, 1983 [26] and Wang, 1996 [27] have shown the limit of this method since the use of the mode conversion reduces the accuracy of the material coefficients. Afterwards, a technique based on the use of direct shear transducers to increase the precision of the measurements was developed Alig, 1997 [28]. This method was then improved in 2000 by Alig, et al [29] where both longitudinal and shear transducers were used. By measuring in a single measurement the longitudinal and
shear properties of the tissuelike materials the acquisition process was made more precise. The use of a direct shear transducer was taken up by Wu, 2002 [30] in which a single shear transducer is used to characterize the shear material properties.

The final aim of this method is to determine the shear properties of soft tissues. To achieve this objective, experimental tests have been conducted firstly on poly(methyl methacrylate) (PMMA) and polydimethylsiloxane (PDMS) with longitudinal transducers to confirm the theoretical work.

5.6 Experiments conducted

Two sets of measurements were performed during our experiments. The first one was to get longitudinal and shear properties as a reference material, which was PMMA, using a basic pulse-echo contact ultrasonic testing. In this case material properties are determined using reflected waves traveling through the PMMA. Then, experiments were performed using the surface reflection method on PMMA and a tissue-like material that were PDMS and brain.

The ultrasonic tests were performed using a square wave generator (Olympus 5077PR) and a 10 MHz longitudinal contact transducer (Panametrics V127). The pulse echo method has been used to calculate the longitudinal wave speed by measuring the time of flight between the second and third reflection. The signals were recorded using a Labview program running on a PXI (National Instrument) and the amplitude and phase of the signal is obtained by the fast Fourier transform. A schematic representation of the set-up is given in Figure 41. A 38 mm thick and 80 mm wide silica block was used as a transmission medium. In fact, due to the comparatively low attenuation of longitudinal
and shear waves in such material the silica block rod can be treated as a lossless medium so that the mechanical impedance is real.

Before each measurement, the electronic devices were switch on 30 minutes before the beginning of the tests in order to warm up the devices. During this time, the equipment and the silica block were cleaned with alcohol, dried, and allowed to reach thermal equilibrium. Then, the signals reflected at the interface silica-air were recorded as a reference. For each of the 7 PMMA samples and for the PDMS sample, 5 signals were recorded in order to calculate the amplitude change and the phase shift. Because of changes of these parameters are often small at least four echoes were taken. The parameters used during all experiments are given in Table 5.

**Table 5: Reflection method parameters.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>PMMA, PDMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (MHz)</td>
<td>10</td>
</tr>
<tr>
<td>Gain (dB)</td>
<td>4</td>
</tr>
<tr>
<td>Pulse Voltage (V)</td>
<td>100</td>
</tr>
<tr>
<td>PRF (HZ)</td>
<td>200</td>
</tr>
</tbody>
</table>

### 5.7 Materials and sample preparation

Experiments were conducted on two materials: poly(methyl methacrylate) (PMMA) and polydimethylsiloxane (PDMS). PMMA is a well-known material for which both longitudinal and shear properties can be easily determined, it was used as reference material to confirm the experimental and theoretical work. In contrast, PDMS is
considered similar to a soft tissue, for which ultrasonic investigations are more difficult using the reflection through the sample.

PMMA is a transparent, tough and rigid plastic produced from the polymerization of methyl methacrylate. The chemical structure of PMMA is shown in Figure 42.

![Chemical structure of PMMA](image)

**Figure 42:** Chemical structure of PMMA [31].

When a high strength is not necessary, PMMA can be used as an alternative to PC. Indeed, it is low cost and it is easy to handle and process making this material good for applications such as swimming pool enclosures, aircraft canopies, instrument panels, and luminous ceilings.

As mentioned, the other material used, as a material similar to soft-tissue, is polydimethylsiloxane (PDMS) a Si based organic polymer. PDMS is the most widely used silicon-based organic polymer, and is particularly known for its unusual rheological (or flow) properties. This material is found in various applications such as contact lenses, medical devices, lubricating oils, and heat-resistant tiles. PDMS is also considered to be inert, non-toxic and non-flammable. The chemical structure of polydimethylsiloxane (PDMS) is shown in Figure 43.
The samples used during experiments were similar to the ones shown in Figure 44 except that they were colorless. It can be seen that this material is relatively flexible.

![Figure 44: Colored PDMS samples.](image)

5.8 Expected results

The theoretical and experimental methods described in the previous parts of this chapter focus on the use of the surface reflection technique to measure the material properties of soft tissues. Tests performed using this method have encountered numerous difficulties and require that more testing on the method be done before using this testing method. As a result, no measurements were obtained using the method that was coherent results.

To help provide a guideline for further study on the method, the following provides the expected results for several materials. These results for reference materials can be used to evaluate the procedures and troubleshoot them. These results are
considered as a first step in a future work in which theoretical and experimental processes will be developed and improved.

The properties of silica block were evaluated using the pulse-echo method for both longitudinal and shear waves using, respectively, 10 MHz and 5 MHz are shown in Table 6.

**Table 6**: Longitudinal and shear properties for the silica block.

<table>
<thead>
<tr>
<th></th>
<th>Longitudinal Waves</th>
<th>Shear Waves</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (MHz)</td>
<td>10</td>
<td>5</td>
</tr>
<tr>
<td>Centered Frequency (MHz)</td>
<td>8.40</td>
<td>4.43</td>
</tr>
<tr>
<td>Wave Speed (m.s(^{-1}))</td>
<td>5965</td>
<td>3766</td>
</tr>
<tr>
<td>Attenuation (Np.m(^{-1}))</td>
<td>19.02</td>
<td>4.42</td>
</tr>
<tr>
<td>Impedance (g.s(^{-1}).cm(^{-2}) x 10^6)</td>
<td>1.313</td>
<td>0.83</td>
</tr>
</tbody>
</table>

Compared to the values of attenuation of soft tissues, for both longitudinal and shear waves, the silica block has a low attenuation. This material should be a good test material since we can ignore the attenuation in the theoretical model.

The silica properties were determined also by using the contact reflection method, considering the setup without a sample. Equation (62) was used to calculate the wave speed for silica blocks with a thickness of 25.4 mm and 38.1 mm. Using the same 10 MHz longitudinal contact transducer the wave speed found for the silica block was 6111 m.s\(^{-1}\) and using the 5 MHz shear contact transducer the wave speed found for the silica
block was 3846 m.s\(^{-1}\). For both longitudinal and shear wave speeds the measurement uncertainty is inferior to be 2.5%.

Using the pulse-echo setup, tests were conducted on PMMA samples. The material properties for both longitudinal and shear waves are found Table 7.

**Table 7**: Longitudinal and shear properties for the PMMA samples.

<table>
<thead>
<tr>
<th></th>
<th>Longitudinal Waves</th>
<th>Shear Waves</th>
</tr>
</thead>
<tbody>
<tr>
<td>Frequency (MHz)</td>
<td>10</td>
<td>5</td>
</tr>
<tr>
<td>Centered Frequency (MHz)</td>
<td>7.60</td>
<td>3.87</td>
</tr>
<tr>
<td>Wave Speed (m.s(^{-1}))</td>
<td>2742</td>
<td>1405</td>
</tr>
<tr>
<td>Attenuation (Np.m(^{-1}))</td>
<td>127</td>
<td>207</td>
</tr>
<tr>
<td>Impedance (g.s(^{-1}).cm(^{-2}) (\times 10^6))</td>
<td>0.323</td>
<td>0.166</td>
</tr>
</tbody>
</table>

Experiments were conducted using contact reflection method on the PMMA and the PDMS, but without reliable results.

### 5.9 Conclusion

For this study Poly(methyl methacrolate) (PMMA) and polydimethylsiloxine (PDMS) samples were studied for measurement of attenuation by reflective contact. The well studied PMMA and a silica block were first tested using standard pulse-echo method. This step is necessary to define the material properties that we then tested using contact-reflection method.
The tests performed with the contact-reflection method were not reliable. This was mainly due to the experimental limitations. Indeed, the accuracy of the measurement is affected by the poor contact between the silica block and the sample.

In this chapter we have presented the idea and the equations for the contact-reflection method that can be used for calculating the properties of soft materials. Theoretically we should be able to obtain values of the properties from the waves reflected from the contact surface without a need for having the wave travel through the unknown sample. This is very attractive for the study of highly attenuating materials such as soft tissue. Even though we have evaluated the expected wave speeds and attenuations for setting up a silica/PMMA interface and using this to measure the properties of PMMA, we could not obtain any consistent results. The results of further study on this method are reported in the master thesis of Jonathan Hein.
Chapter 6

SUMMARY AND CONCLUSION

The focus of this thesis was to study and understand, through different ultrasonic methods, the characteristics and structure of complex materials. These include plastically deformed and aged polycarbonate, a structured PC-like plate with water filled cavities, bovine bone, and a PDMS used as a skin simulant.

We conducted ultrasonic wave speed measurements on plastically compressed samples with 25% and 50% plastic engineering strains. These samples were aged for various aging times up to 6000 hours at 105° C, or up to 600 hours at an aging temperature 125° C. In order to evaluate if ultrasonic measurements could be used to evaluate the observed loss in toughness as a result of thermal aging, the results were compared with the Charpy tests results done by Strabala (2009) and Meagher (2010). The drop of toughness observed in Charpy test results for the 25% TA and TT oriented samples at both 105° C and 125° C were not noticed in the ultrasonic measurements. It
was concluded that ultrasonic measurements alone cannot be used to determine the loss in toughness.

With the hope to develop a synthetic material which could mimic the material behavior of bone, pseudo-PC appears to be an interesting alternative. Seven different structures were constructed by changing the density, the dimensions and the orientation of channels manufactured in a plate of the pseudo-PC. It was demonstrated that the percentage of volume occupied by the channels had the most influence on the wave speed. Indeed, the wave speed decreases from 2580 m.s\(^{-1}\) for the pure pseudo-PC to 2170 m.s\(^{-1}\), which is nearly 40%. The effect of the structure on the attenuation is also observed but the variation of the attenuation can’t be easily associated with the structure. The other point is that for a thickness relatively close to human bone skull, this work has shown that a synthetic material could be developed with the aim of mimicking the bone.

Ultrasonic measurements performed on bone have shown coherent results with the pulse-echo method. Measurements of the wave speeds and also of the first critical angle were possible because of the compact structure of the bone samples studied. The ultrasonic setups used to investigate the shear wave speed and material structure were not accurate enough to give reliable results.

Ultrasonic inspections of soft tissues are made difficult due to the high attenuation of such materials using pulse-echo method. To characterize the shear properties a contact-reflection technique is required. From an experimental point of view, preliminary tests have shown that the measurements are not repeatable in the current state. Theoretically we should be able to obtain values of the properties from the waves
reflected from the contact surface without the need for having the wave travel through the unknown sample. The study that has been done is considered as preliminary work. This is why more tests have to be performed to get reliable results.

6.1 Future work

Several topics have been identified for future work. An ultrasonic wave speed study could be conducted for several other aging times on the 25% plastic compressed PC samples to confirm the lack of correlation between these tests and Charpy tests at 105° C and 125° C aging temperatures.

For the structured pseudo-polycarbonate samples, the very first step is to compare the future results performed on bone or skull before constructing or improving current sample structures. In addition, this technique could be use with other polymers such as the ones presented Table 8. Indeed, plastic materials, besides their intrinsic properties, have a large range of densities and wave speeds which could be used to develop new synthetic materials and thus more biological materials could be mimicked.

Table 8: Acoustic Properties of Polymers [1].

<table>
<thead>
<tr>
<th>Polymer</th>
<th>Longitudinal Velocity (m.s(^{-1}))</th>
<th>Density (g.cm(^{-3}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>PC</td>
<td>2200</td>
<td>1.2</td>
</tr>
<tr>
<td>PMMA</td>
<td>2740</td>
<td>1.18</td>
</tr>
<tr>
<td>PVC</td>
<td>2395</td>
<td>1.4</td>
</tr>
<tr>
<td>PS</td>
<td>2670</td>
<td>1.06</td>
</tr>
<tr>
<td>PE</td>
<td>1940</td>
<td>0.9</td>
</tr>
</tbody>
</table>
About the work on bone, tests could be performed on bone and in particular human skull bone using the through transmission method. The results obtained in this study clearly show that the experimental setup needs to be improved in such way that the parameters and the environment are controlled to insure accurate results. This requires accurately aligning the immersion transducers in addition to the location of the focus length between them. Parameters such as the scattering could also be studied. By performing the polar through transmission results and by studying the backscattering information, one can study the microstructure. This technique is based on the fact that both the transmitter and the receiver are rotated symmetrically in relation to the bone sample. This setup is used to observe the backscatter since there is no front or back wall in such a configuration.

About the ultrasonic tests on soft tissues, in addition to PMMA, tests could be conducted on other well studied materials such as PC. In this way the theoretical model could be tested. In addition, the experimental approach could be improved to limit the error in the measurements. This could be done by improving the contact surface between the silica block and the sample or by adding a glass plate on the silica block, which will provide multiple echoes which may increase the precision.
References


