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**Resonant Ultrasound Spectroscopy**

Analysis of thickness resonances to characterise a material

Master Thesis Project

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1. **Introduction**

The aim of this project was to establish reliable setup to characterise a living material using resonant ultrasound spectroscopy. In the first stage of the project, the solid materials such as aluminium, glass and plastics were tested to prove that setup is well tested and ready to switch to a living materials such as plants. The experiments were performed with aluminium, glass and plastic due to existing elastic constants, hence it is straightforward to compare datasheet values to analysed data.

The main reason why exactly ultrasound resonant spectroscopy was used is that that by measuring transmission coefficient of a sample, RUS can infer parameters like thickness, density, attenuation coefficient, speed of sound and first order resonant frequency. Moreover, this technique is non-destructive, non-invasive, rapid and relatively inexpensive. Therefore, there is no damage on a sample which is significantly important for a plant samples, while running the experiment.

Coefficient of transmission of sound at normal incidence through each sample in the frequency range 0.6 MHz – 1.7 MHz was measured. For all cases, at least one thickness resonance was observed. From these measurements density, sound velocity, and attenuation of ultrasonic longitudinal waves were obtained and compared to available data provided in an articles or by manufacturers.

The method is based on frequency-domain analysis, by using the Fast Fourier transform, of pulse transmitted through a sample.

All the experiments were conducted in Biology department of Albert-Ludwig’s University of Freiburg city in Germany. And the required equipment, except oscilloscope (listed below) were transported from Heriot-Watt University, Edinburgh:

* Transducer
* Hydrophone
* Water tank
* Pulser/receiver
* DC coupler
* Oscilloscope

Results reveal that these resonances are strongly sensitive on different parameter changes which are discussed further in the report.

This report goes through all the steps and problems which are faced during the experiments and gives suggestions for further improvements.

All the needed information such as analysed data, Matlab and Python codes, setup instruction, the report can be provided via GitHub control system.

1. **Literature review and theory**
   1. **Fundamentals of ultrasound**

**2.1.1. Ultrasound**

The sound waves with frequencies above 20 kHz is called ultrasound and they are not in range for human hearing (William, 2012). Ultrasound is broadly used technique in different applications of medicine, food industry, factories and non-destructive testing. Sending and receiving of transmitted or reflected ultrasonic pulses allows ultrasonic devices to detect objects, defects and measure distances.

Ultrasound imaging (sonography) is mostly used in medicine to identify a health or gender of baby. In the non-destructive testing of materials or structures, ultrasonic waves are used to detect flaws. Industrially, ultrasound is used for cleaning, mixing and to accelerate chemical processes. In living environment, animals like bat and porpoises use ultrasound to locate prey and obstacles.

**Table I.** Frequency classification of Ultrasound

|  |
| --- |
| Frequency (Hz) Classification |
| 20 – 20.000 Audible sound  20.000 – 1.000.000 Ultrasound  1.000.000 – 30.000.000 Diagnostic ultrasound |

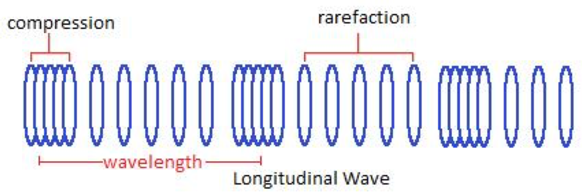
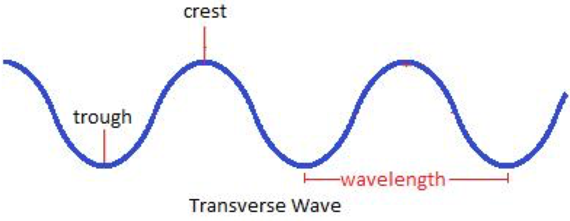
**2.1.2. Ultrasonic waves**

In ultrasound, high-frequency sound waves travel through the material being tested and information about parameters of a material are then obtained by measuring the type and interaction angle between the sound wave and the testing sample. When the sound waves are introduced within a sample, alternating molecular compression and rarefaction takes place. There are 2 modes of waves which propagates through a solid material:

* Longitudinal waves (pressure waves)
* Transverse waves (shear waves)

Longitudinal waves, where oscillation happens in the same direction as the wave is moving. This type of wave can be generated in liquids, solids and gases. In transverse wave, the oscillation occurs perpendicular to the direction that the wave is travelling in. This type of wave is propagated in solid structures only. Figure 1 represents these types of waves.

**Figure 1.** Longitudinal and transverse waves (http://www.keywordsuggests.com/)

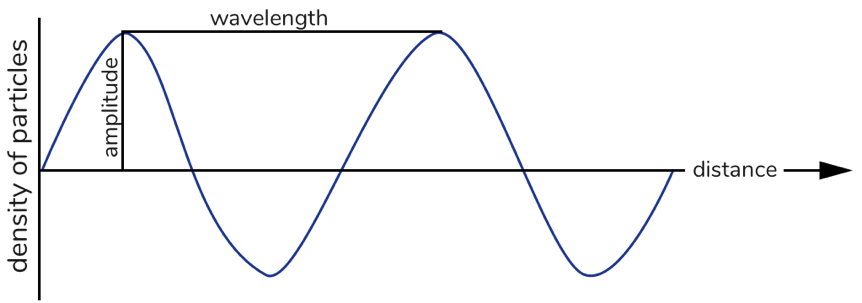
 

**Wave characteristics**

An area of compression and a neighbouring zone of rarefaction identify one cycle of an ultrasound wave. A wave cycle can be depicted as a graph of local pressure in the medium versus distance I along the direction of the wave (Figure 2). The wavelength is the distance covered by one cycle. The number of cycles per unit time introduced in the medium each second is referred to as the frequency, and measured in unit of hertz, kilohertz or megahertz, where 1 Hz is 1 cycle per second. The maximum height of the wave cycle is referred to amplitude of the ultrasound wave. And the multiplication of the frequency () and the wavelength (λ) is the velocity of the wave and expressed as below (William, 2012):

(1)

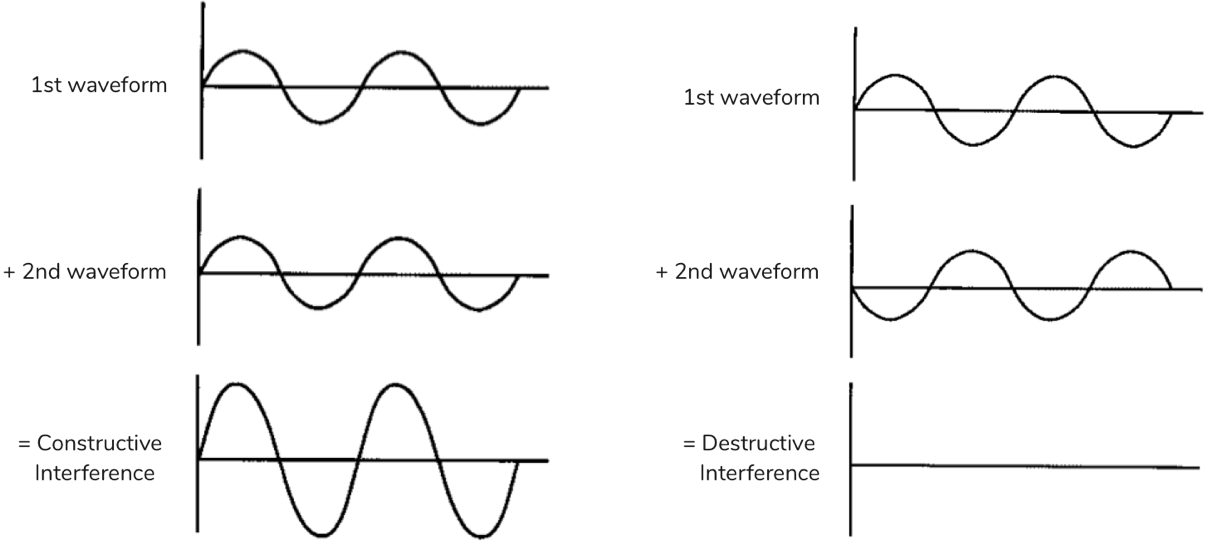
**Figure 2.** Characteristics of an ultrasound wave



**Interaction of waves**

Interference of waves occurs when to waves meet. There are two extremes of waves’ interference: constructive and destructive. In constructive interference peak meets with other peak, they are said to “in phase” and in destructive interference the waves are “out of phase”, hence two waves cancel each other out. Waves experience constructive interference when their amplitudes added, whereas waves undergoing destructive interference can completely nullify each other (Figure 3).

**Figure 3.** Interference of two waves



**Reflection and Transmission**

The part of incident energy reflected from the surface directly depends on the different in acoustic impedance of the material on opposite sides of the interface. The acoustic impedance can be expressed as follows:

(X)

where Z is acoustic impedance of medium, is density of the medium and is the speed of sound in the medium, is angular frequency and is wavenumber (explained below).

In a case of one layer material the reflection coefficient is shown below (ultrasound wave incident perpendicular):

(X)

where Z1 and Z2 are the acoustic impedances of two different media. The fraction of the incident energy is transmitted through the media and can be described by transmission coefficient:

(X)

Therefore, it is clear, that:

(X)

A large impedance mismatch occurs at an interface, when the most of energy if reflected, and only small portion is transmitted across the interface. For instance, ultrasound energy is significantly reflected at air-

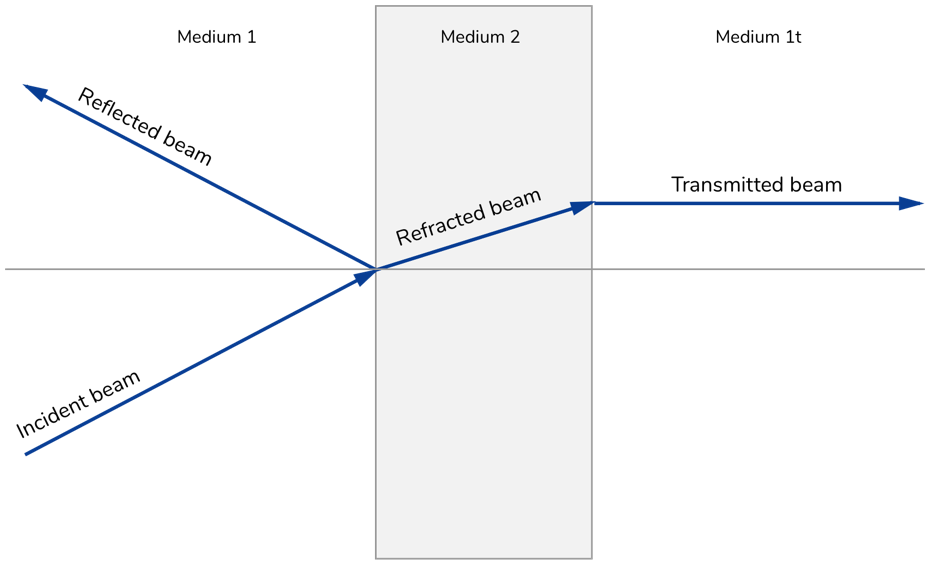
tissue and air-water interfaces, because the air impedance is much less than tissue or water.

**Refraction**

The direction of incident beam is changed once it crosses an interface obliquely between two media. If the velocity of ultrasound is higher in a second medium, then the beam enters this medium at less steep angle. This behaviour of ultrasound beam is called refraction. The relationship between incident angle and refracted angle can be described by Snell’s law:

(X)

**Figure 4.** Reflection, refraction and transmission of ultrasound



**Note to Figure 4:** The beam hits the medium 2 at an angle of . A portion of the energy is reflected at an angle of and part of energy goes through at an angle of .

**2.1.3. Ultrasound intensity**

As an ultrasonic waves pass through a medium, it transfers energy through the medium. The amount of energy transport is called “power”.

The rate of flow of energy (power) per unit of cross sectional area is called intensity. Intensity is commonly described relatively to another intensity; for example, the intensity of ultrasonic waves transmitted through medium may be compared with that of the ultrasound sent into the material. The intensity is measured in a logarithmic scale, since it is the most appropriate for recording data over a range of many orders of magnitude. The decibel scale is used in acoustics:

(X)

where is the reference intensity. Due to intensity is power per unit area and power is energy per unit time, it is possible to write above expression as:

(X)

Ultrasound wave intensity is allied to maximum pressure () in the medium by the following expression:

(X)

where is the density of the medium and is the speed of sound in the medium. When we substitute Eq. (X) for and in Eq. (X):

(X)

While comparing two pressure waves, Eq. (X) can be used directly; the pressure does not have to be converted into intensity to find dB value.

**2.1.4. Ultrasound velocity**

The speed of an ultrasonic wave through a medium changes and it depends on the physical properties of the medium. The velocity of an ultrasonic wave is relatively low in low-density media such as air and gases, since the molecules in them move over relatively large distances before they impact neighbouring molecules. In solids, the molecules are limited in their motion, and the ultrasound velocity is relatively high. In

another hand, liquids demonstrate ultrasound velocities in-between those, gases and solids.

**Table II.** Approximate velocities in different medium

|  |  |
| --- | --- |
| Nonbiological material Velocity (m/s) | Biological material Velocity (m/s) |
| Aluminium 6400  Plastic 2680  Water (degassed) 1480  Glass 5640  Air 330 |  |

**2.1.5. Ultrasound attenuation**

As an ultrasound beam penetrates a medium, energy decreases due to absorption, reflection and scattering, so to say, the attenuation of ultrasound is when sound intensity decreases exponentially with distance from the source.

Donation to attenuation of an ultrasound beam can include:

* Reflection
* Absorption
* Scattering
* Refraction
* Diffraction
* Interference

Ultrasound energy is absorbed by a medium when the fraction of the beam’s energy turned into other forms energy, like an increase in the arbitrary motion of molecules. Ultrasound is reflected back when the angle of reflection is same as incident angle. If the part of beam changes direction in a less orderly manner, this phenomenon is called “scattering”.

The attenuation of ultrasound in a medium is expressed by attenuation coefficient α in units of dB/cm or Nepers per meter(1 Np = 8.686 dB). The attenuation coefficient is the total amount of individual coefficients for scattering and absorption and can be expressed as follows:

(X)

where α is attenuation coefficient, A is amplitude of the ultrasound wave, A0 is the initial amplitude and is the distance the wave has travelled through the medium. (McClements and Gunasekaran, 1997)

**Table III.** Attenuation coefficient for 1 MHz ultrasound

|  |
| --- |
| Medium α (dB/cm) |
| Blood 0.18  Fat 0.16  Muscle 3.3  Water 0.0022  Bone 20  Brain 0.85 |

**Note to Table III:** The given figures in table are relative and may vary with both the origin and condition of the biological sample.

* 1. **Ultrasound transducers**
     1. **Introduction to transducers**

A transducer is certain device that transforms one form of energy into another. And ultrasound transducer converts electrical energy into ultrasound energy and in reverse way. Transducer for ultrasound consists of piezoelectric crystals or elements such as quartz (silica).

* + 1. **Piezoelectric effect**

A piezoelectric effect is exhibited whenever a pressure is applied, develop voltage across opposite surfaces. This effect is used to produce ultrasound incident wave when electrical signal is applied to the transducer. Application of the voltage across the crystal causes its deformation – expansion or compression depending upon voltage polarity.

A definition of transducer is known as a fraction of applied energy that is converted into wanted energy mode. For ultrasound transducer, this definition described with electromechanical coupling coefficient; it varies whether electrical or mechanical energy is applied:

If electrical energy is applied:

Some values for transducers are listed below in Table IV

**Table IV.** Properties of piezoelectric crystals

|  |
| --- |
| Materials Electromechanical coupling coefficient () |
| Quartz 0.11  Rochelle salt 0.78  Barium titanate 0.30  Lead zicronate titanate (PZT-4) 0.70  Lead zicronate titanate (PZT-5) 0.70 |

* + 1. **Transducer design**

The piezoelectric crystal is essential part of any transducer. A crystal generates its greatest response at resonant frequency. The resonance frequency of ultrasound transducer depends on the thickness of crystal. As the crystal goes through one

complete cycle form shrinking to expansion to the next shrinking, compression waves move in direction of centre of the crystal form opposite side of its face. It is complicated to “drive” crystal with one wavelength thickness due to the compression waves arrive at opposite surfaces just as the next shrinking starts, hence the energy is wasted. However, if the thickness of crystal is equal to half of wavelength, a compression waves happens at the crystal interface just as expansion begins to occur. A crystal with half wavelength resonates at a frequency:

(x)

The plastic sealing of crystal has a thickness of ¼ and it is called quarter-wavelength matching layer.

**3.2. Results and discussion**

As stated in introduction section, the purpose of project was to establish reliable setup to have reproducible and repeatable data. The experimental setup is straightforward and data analysis process is fast and simple. However, since there is large amount of parameters there were certain issues regarding the equipment and the data analysis process. They were tested accordingly in order to investigate how the changes of different parameters can influence on data results. All the measurements were performed with solid materials, mostly aluminium sample. The reason behind of experimenting on solid materials is it is simple to compare the retrieved results with literature values or data sheet. That proves that the setup and technique is functioning in correct way and data is reproducible before moving into biological species. The subsections below will show how the results change according to variation of parameters.

**3.2.1 Measurements with samples on default settings**

Before understanding the variation of parameters, the default settings for pulser/receiver and oscilloscope (Frederike, 2016) were used to obtain and compare the results afterwards. The experiments were conducted only on the samples which were available at that time with different thicknesses to see the results and continue ….. Default settings are depicted in a Table X below:

**Table X.** Settings for pulser/receiver and oscilloscope by default

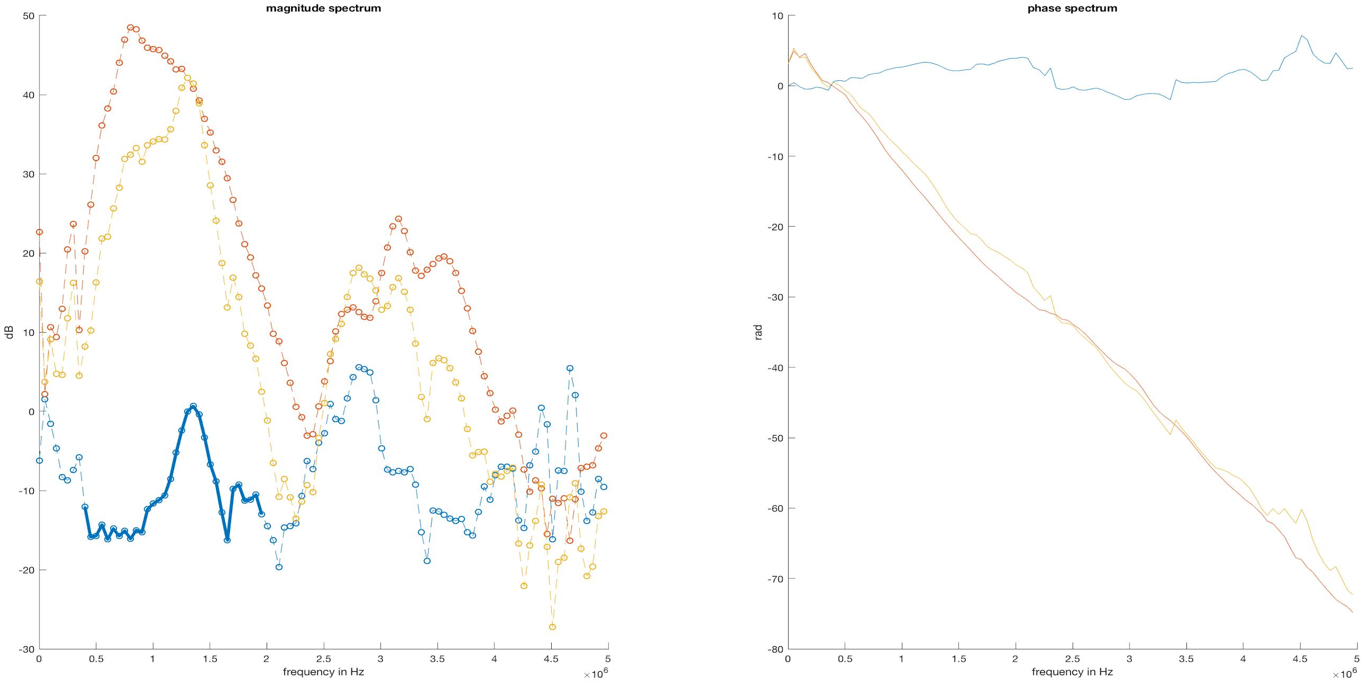
|  |  |
| --- | --- |
| Pulser/receiver | Oscilloscope |
| Relative gain 38 dB  High pass filter out  Low pass filter 35 dB  Pulse repetition frequency: 1 (100Hz)  Pulse amplitude 11  Trigger Internal (INT)  Pulse energy High 3  Damping 2 | Amplitude 5 V/division  Time 2μs/division  Delay 72.56 μs |

All the lines represented in pictures:

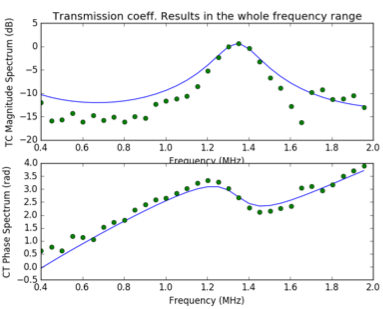
* Red line on the graphs is measured data without sample
* Yellow line on the graphs is measured data with sample
* Blue line on the graphs is the difference between measured data with sample and without sample
* Section with bold blue line on magnitude graphs is the measured data which is process in python

**Experiment 1. Glass with 2 mm thickness**

**Figure X1.**  Detail in the phase and magnitude spectrum of the first received pulse without and with sample between transducer and hydrophone. The graph shows MATLAB output

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**Figure X2.**  Transmission coefficient spectra with fitted theoretical transmission coefficient of a measured glass sample.

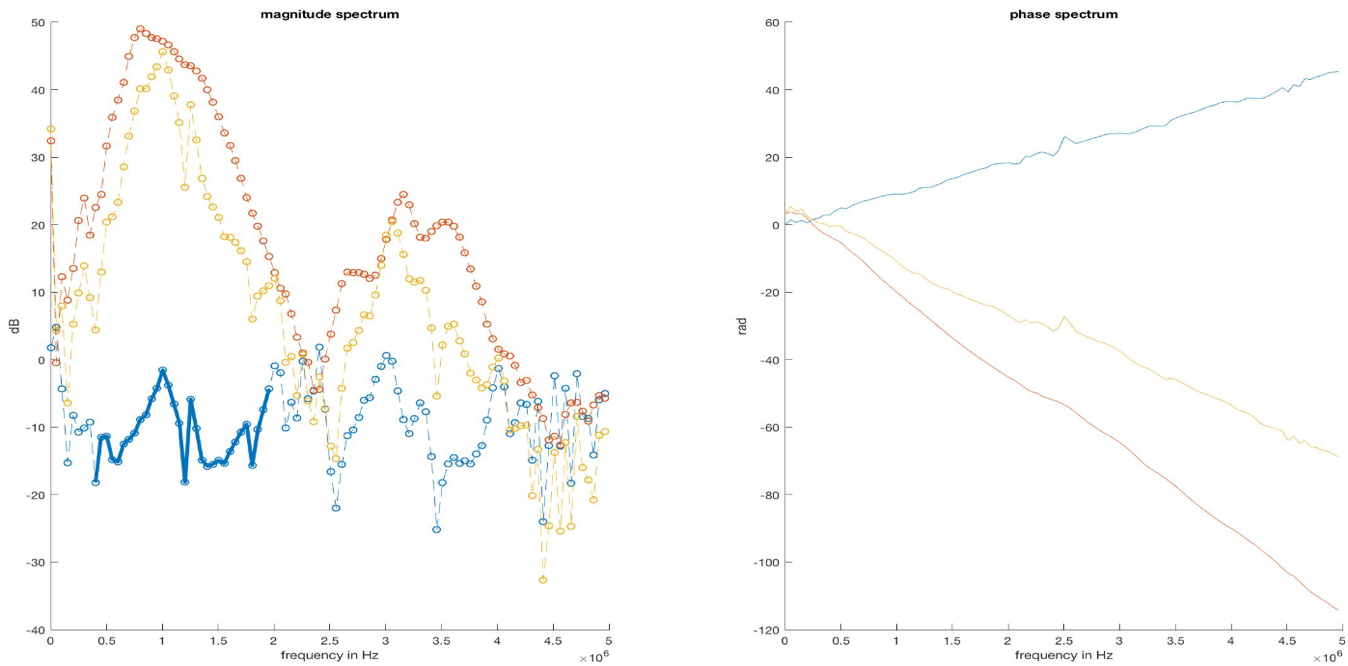
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**Table X1.** The results obtained from python algorithm compared with calculated and/or literature data for further investigation on the issues of setup and/or data analysis.

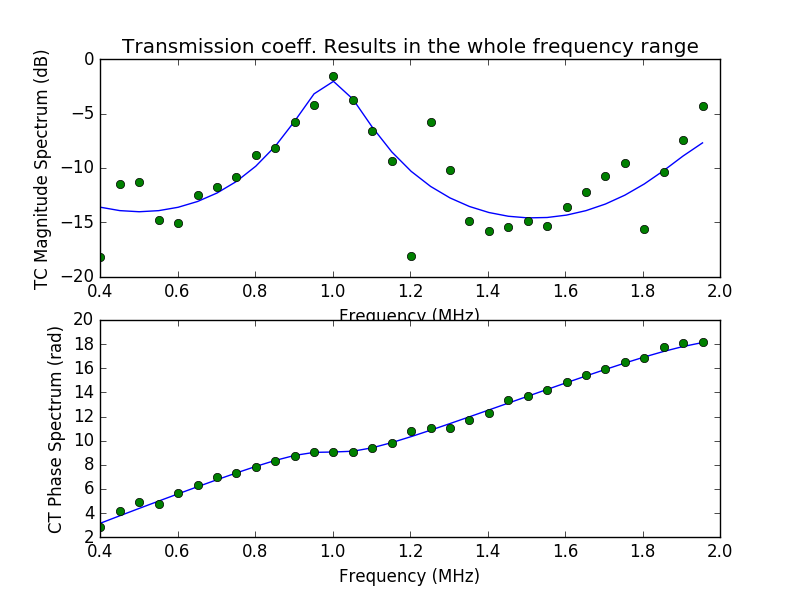
|  |  |
| --- | --- |
| Parameters Python output | Calculated and/or literature data |
| Thickness (mm 0.98  Velocity (m/s) 2726  Density (kg/m3) 4293  Resonant frequency (MHz) 1.35 | 1.92  4540  2719  1.18 |

**Experiment 2. Glass with 3 mm thickness**

**Figure X2.** Detail in the phase and magnitude spectrum of the first received pulse without and with sample between transducer and hydrophone. The graph shows MATLAB output

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**Figure X3.**  Transmission coefficient spectra with fitted theoretical transmission coefficient of a measured glass sample.

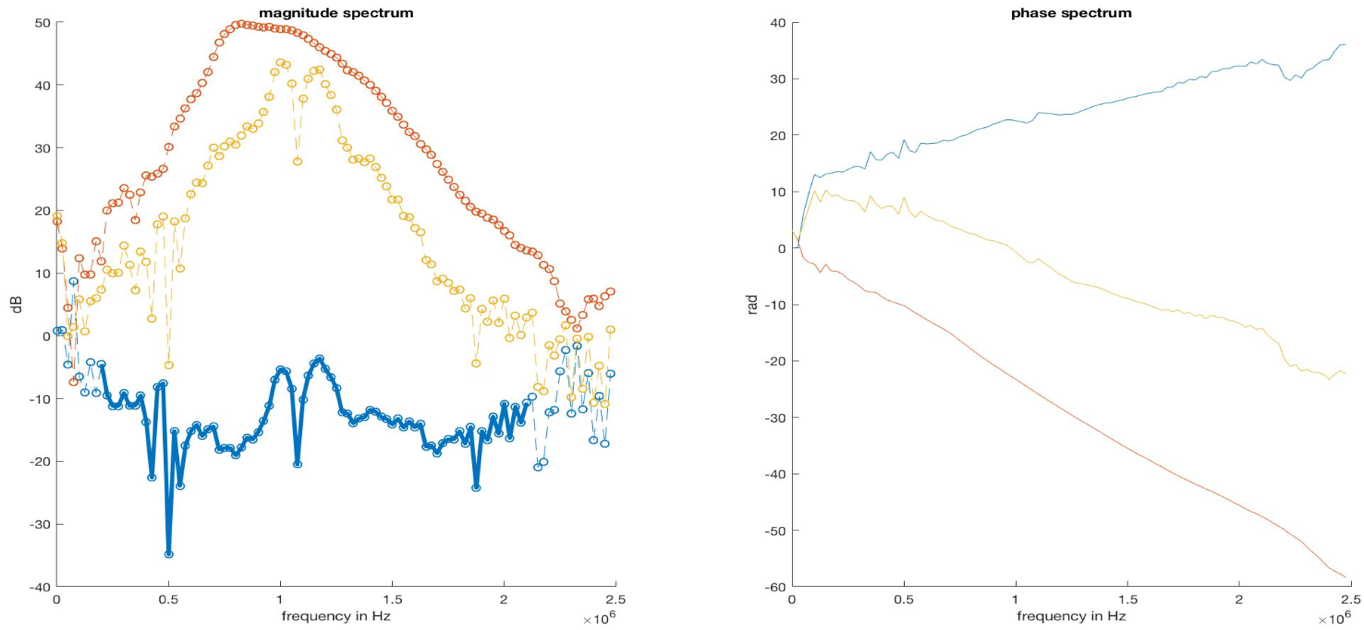
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**Table X2.** The results obtained from python algorithm compared with calculated and/or literature data for further investigation on the issues of setup and/or data analysis.

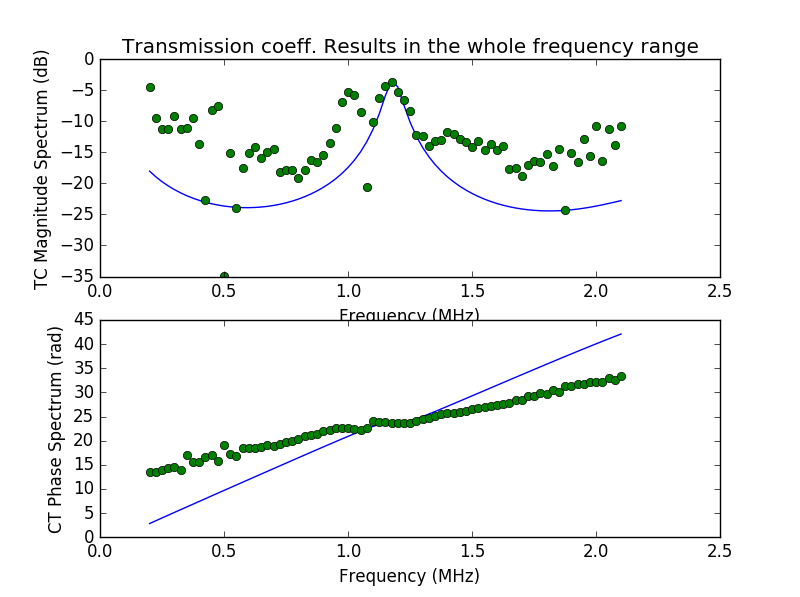
|  |  |
| --- | --- |
| Parameters Python output | Calculated and/or literature data |
| Thickness (mm 2.80  Velocity (m/s) 5662  Density (kg/m3) 2596  Resonant frequency (MHz) 1.00 | 2.72  4540  2683  0.84 |

**Experiment 3. Aluminium with 3 mm thickness**

**Figure X5.**  Detail in the phase and magnitude spectrum of the first received pulse without and with sample between transducer and hydrophone. The graph shows MATLAB output



**Figure X6.**  Transmission coefficient spectra with fitted theoretical transmission coefficient of a measured glass sample.



**Table X3.** The results obtained from python algorithm compared with calculated and/or literature data for further investigation on the issues of setup and/or data analysis.

|  |  |
| --- | --- |
| Parameters Python output | Calculated and/or literature data |
| Thickness (mm 5.10  Velocity (m/s) 12186  Density (kg/m3) 3677  Resonant frequency (MHz) 1.17 | 2.76  6400  2830  1.16 |

As it can be seen from results above, it did not fit nicely with expected values. However, it showed acceptable correct resonant frequencies. First experiments with affordable samples gave a common understanding emerged on the need for further research with aluminium samples with different thicknesses which are manufactured in workshop later. Further in this section the focus was on improving this results in varying degrees. The first assumption was to guarantee that the equipment, specifically pulser/receiver is working as intended, since the pulser/receiver amplifies and enhance signal-to-noise to produce clear signal on the scope. A number of experiments on pusler/receiver were performed to select an optimal option as a new default setting.

**3.2.2. Characterisation of pulser/receiver**

The figure below depicts a front panel of pulser/receiver and shows all-wheel control button which are available on it.

**Figure X.**  The front panel of pulser/receiver indicating all the available switches on it.

