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Young's Modulus Determination of Polyester and Epoxy by Means of Ultrasonic Pulse Echo Testing

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Abstract. The Young's modulus or elasticity modulus is material ability to resist elastic deformation when loaded. Thermosetting polymers have a wide range of modulus elasticity values. To determine the elasticity modulus value, materials need to be destructive tested, such as tensile test or bending test. But in industrial field, they avoided destructive test. Theoretically, the modulus of elasticity can also be determined by ultrasonic testing pulse echo method. This method has already applied successfully for metals material. The goal of this research was to evaluate probe used in longitudinal velocity measurement of polyester and epoxy and to determine Young's Modulus of polyester and epoxy by means ultrasonic pulse echo testing. This method was performed using three longitudinal probes that have different diameters, frequencies, and additional delay line. The specimens were varied with different thicknesses. The ultrasonic pulse echo method uses probe 3 with a diameter of 12.7mm, a frequency of 1.5MHz, and additional delay line is most suitable to measure the longitudinal velocity of polyester and epoxy with thickness of 2.82-36.72 mm. Probe 3 has a smaller diameter and additional delay lines thus eliminating the effect of dead zones. The elastic modulus error of thermoset polymer material through ultrasonic testing of pulse echo method compared to the mechanical modulus of elasticity is still very large, i.e. 102% for epoxy, and 159% for polyester. A large error is caused by attenuation and the use of the elasticity modulus equation from ultrasonic testing that still requires modification.

Keywords: Elastic Modulus, Ultrasonic Testing, Pulse Echo, Polyester, Epoxy, Longitudinal Velocity



1. Introduction

Thermosetting polymers, e.g. unsaturated polyesters and epoxies, are widely used in industries. Unsaturated polyesters are extensively used to produce structural parts with a glass-fiber reinforcement, while epoxies are employed for aeronautical and electronic applications [1]. Thermosetting polymers have a wide range of Young's modulus values which affected by its structure and dependent on molecular weight, molecular orientation, and density [2]. Young's modulus, also known as elastic modulus, is an important mechanical property of materials. This property describes the stiffness of materials or material's resistance to elastic deformation [3]. The elasticity of thermoset polymer elasticity can decrease due to degradation. This degradation generally caused by components exposed to ultraviolet light, high temperatures, and chemical liquids so that the polymer chain chain begins to break [4]

To determine the elastic modulus, materials need to be tested destructively, such as tensile test or bending test. Industrial field tries to avoid destructive tests as it will damage the test object and cannot be conducted in-situ. Ultrasonic testing is common non-destructive test technique that frequently used to detect defects in materials [5]. The modulus of elasticity theoretically can be determined by ultrasonic testing. Ultrasonic wave propagation in material has strong proportional relation to its elastic properties [6].

Two common methods of ultrasonic testing are pulse echo and through transmission [7]. Ultrasonic through transmission testing for measurement of polymers' elastic modulus and dynamic elastic modulus have been studied, however this method is not practical as it requires two sides of the test object, and immersed in liquid [8]. Ultrasonic pulse echo testing is more practical to be used in industries, especially in the real time monitoring of in service structures as it only requires one side of the test object. Determination of Young's modulus with ultrasonic testing has already applied successfully for metals with error less than 5% [9].

Error in determination of Young's modulus of polymers by means of ultrasonic testing can be caused by some factors such as attenuation and properties of materials i.e. density and viscoelasticity [9]. Attenuation is a factor that describe the decrease in ultrasound intensity with distance, normally expressed in decibels per unit length [10]. When sound travels through a medium, its intensity diminishes with distance, or in the other hand, material thickness. In idealised materials, sound pressure as signal amplitude is only reduced by the spreading of the wave. Ultrasonic attenuation is the decay rate of the wave as it propagates through material [11]. Higher attenuation experienced by material can caused higher error in Young's modulus determination by ultrasonic testing also higher viscoelasticity of material. While higher density of material reduced error in Young's modulus determination with ultrasonic testing [12].

Dead zone and near field common phenomenon occurred in ultrasonic testing. Both will made difficulties in ultrasonic testing reading. Dead zone is the distance in material from the surface of the test specimen to the depth at which a reflector can first be resolved under specified conditions [5]. Length of dead zone depends on probe and ultrasonic test equipment. Meanwhile near field is the distance immediately in front of the transducer in which the ultrasonic beam exhibits complex and changing wave fronts [10].

To improve the inspection with ultrasonic testing changing frequency and diameter of the probe can be done. Also a zero impedance probe (ZIP), probe with delay line is commonly used. The primary function of a delay line is to introduce a time delay between the generation of the sound wave and the arrival of any reflected waves. This allows the probe to complete

its "sending" function before it starts its "listening" function so that near surface resolution is improved [13]. These will eliminate or reduce dead zone or near field factor.

Therefore, this research aims to evaluate the use of different probes to determine longitudinal velocity of polyester and epoxy and to determine Young's Modulus of polyester and epoxy by means ultrasonic pulse echo testing.

2. Materials and Methods

The polyester specimens were made from unsaturated polyester resin Yucalac ® C108-B with 2% weight curing agent. Eposchon A as primer epoxy resin and Eposchon B as hardener used to make the epoxy specimens with 1:1 weight ratio. Both thermosets were cured in room temperature. The thickness of polyester ultrasonic specimen used for the experiment are 2.8 mm, 11.6 mm, and 36.7 mm. While the thickness of epoxy specimens are 3.9 mm, 13.2 mm, and 33.7 mm. Material density measurement was conducted with KERN ABJ-NM/ABS-N device at 25°C using water media according to ASTM D 792 [14].

Ultrasonic test was conducted with GE USM 35X device with Pulse-Echo method to obtain the longitudinal wave velocity of materials. Sonotech® Ultragel II® was used as the couplant. Both showed in Figure 2.1.

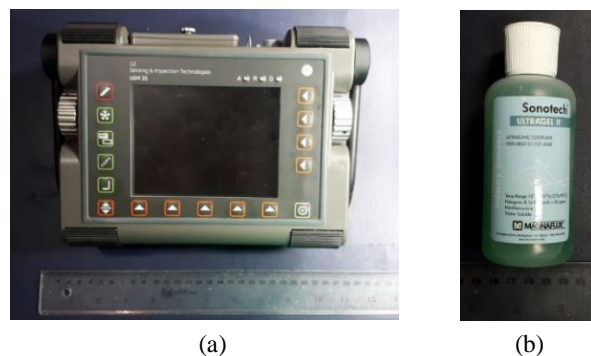
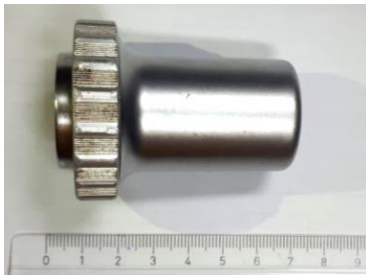
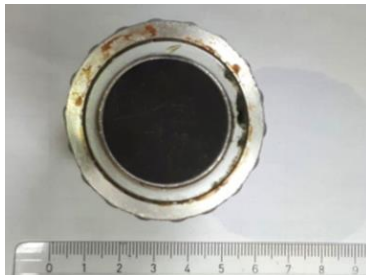
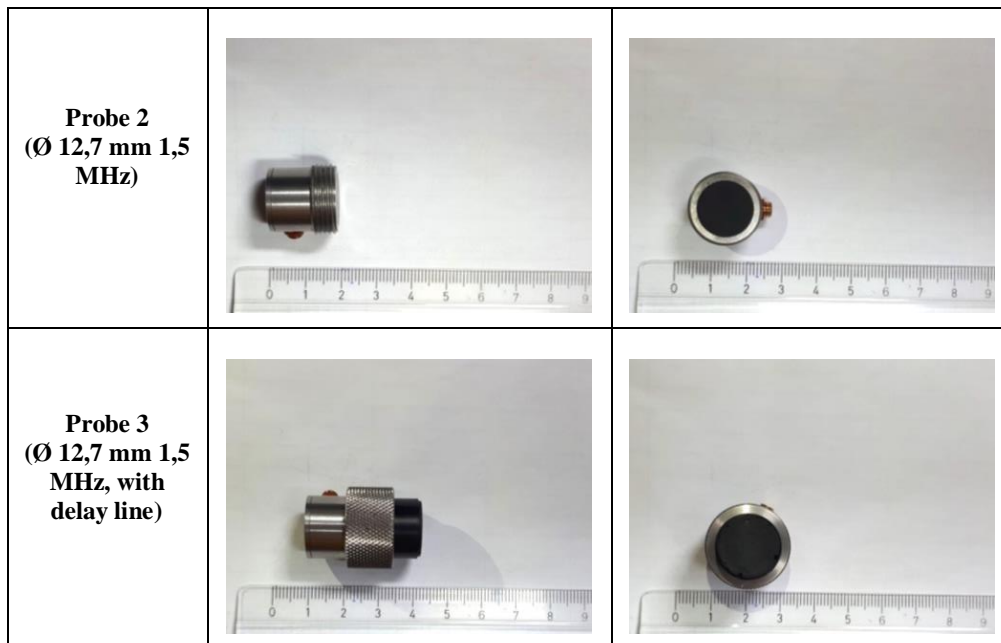


Figure 2.1 (a) Ultrasonik device GE USM 35X and (b) couplant

There were 3 probes used for the ultrasonic test: probe 1 using 1 MHz frequency with 25.4 mm diameter; probe 2 using 1.5 MHz 12.7 mm; and probe 3 using 1.5 MHz 12.7 mm with 13 mm thickness delay line. Table 2.1 exhibit the probes.

Table 2.1 Probes

Probe	Side view	Below View
Probe 1 (Ø 24 mm 1 MHz)		



Three times of longitudinal wave measurement in every section were conducted on polyester and epoxy by means of all three probes. The velocity is calculated based on ASTM E 494 [15]. The longitudinal wave velocity obtained from the measurement was used to obtain Young's modulus using **Equation 1**.

$$E = \frac{V_{long}^2 \times \rho \times (1 + \nu)(1 - 2\nu)}{(1 - \nu)} \quad (1)$$

where V_l is longitudinal sound velocity, ρ is material density, E is modulus of elasticity, and μ is material Poisson's ratio.

The apparent attenuation (α) is determined by means of **Equation 2**, based on ASTM E 664 [16]. 36.72 mm polyester and 33.71 mm epoxy were used to obtain apparent attenuation for each material.

$$A = A_0 \times e^{-\alpha z} \quad (2)$$

where A the amplitude that is reduced after the wave has passed the distance z , A_0 is initial amplitude, α is attenuation coefficient, and z is distance travelled.

Tensile test was conducted to obtain the elastic modulus of polymeric specimens based on ASTM D 638 [17]. The specimens were type I for dimension with speed of testing was 5 mm/min. The Young's modulus obtained from tensile test shall be the reference and will be compared with the Young's modulus resulted from the ultrasonic test. Poisson's ratio of polyester and epoxy were from literature [18].

3. Results and Discussion

3.1 Physical Properties

Table 3.1 presents the average density of polyester used was 1.21 g/cm³ with poisson's ratio value was 0.36. Meanwhile epoxy has 1.10 g/cm³ density and 0.37 poisson's ratio rate. The densities achieved in this experiment within range of literature's density [19]. Poisson's ratio literature was used in this research.

Table 3.1 Polymers Density and Poisson's Ratio

Polymers	Average Density (g/cm ³)	Poisson's Ratio
Polyester	1.21	0.36 [18]
Epoxy	1.10	0.37 [18]

3.2 Probes Evaluation Results

Table 3.2 and **Figure 3.1** depicts the longitudinal velocity of polyester and epoxy specimens obtained from ultrasonic testing with different thicknesses and probes compared with literature [18, 20]. The measurement of longitudinal velocity using the third probe that used delay line, obtain better results for thin specimens (2.8 mm thickness polyester and 3.9 mm epoxy) than the other probes. The result of longitudinal velocity measurement using of delay line tend to be have stable values for each polymers, though there were thickness differences, unlike the first and second probe that did not use delay line material.

Table 3.2 Longitudinal velocity measurement result for polyester and epoxy with specimens thickness variation using different probe

Polymers	Specimen Thickness (mm)	Ultrasonic longitudinal velocity				
		Experiment			References	
		Probe 1 (1 MHz Ø 24 mm)	Probe 2 (1,5 MHz Ø 12,7 mm)	Probe 3 (1,5 MHz Ø 12,7 mm with delay line)	Lower limit	Upper limit
Polyester	2,84	1831±23	1904±11	2505±59	2290	2430
	11,64	2416±78	2367±27	2540±15		
	36,72	2518±26	2501±21	2551±14		
Epoxy	3,92	2146±22	2122±4	2306±16	2480	2680
	13,24	2228±61	2214±7	2330±12		
	33,71	2344±1	2297±24	2336±61		

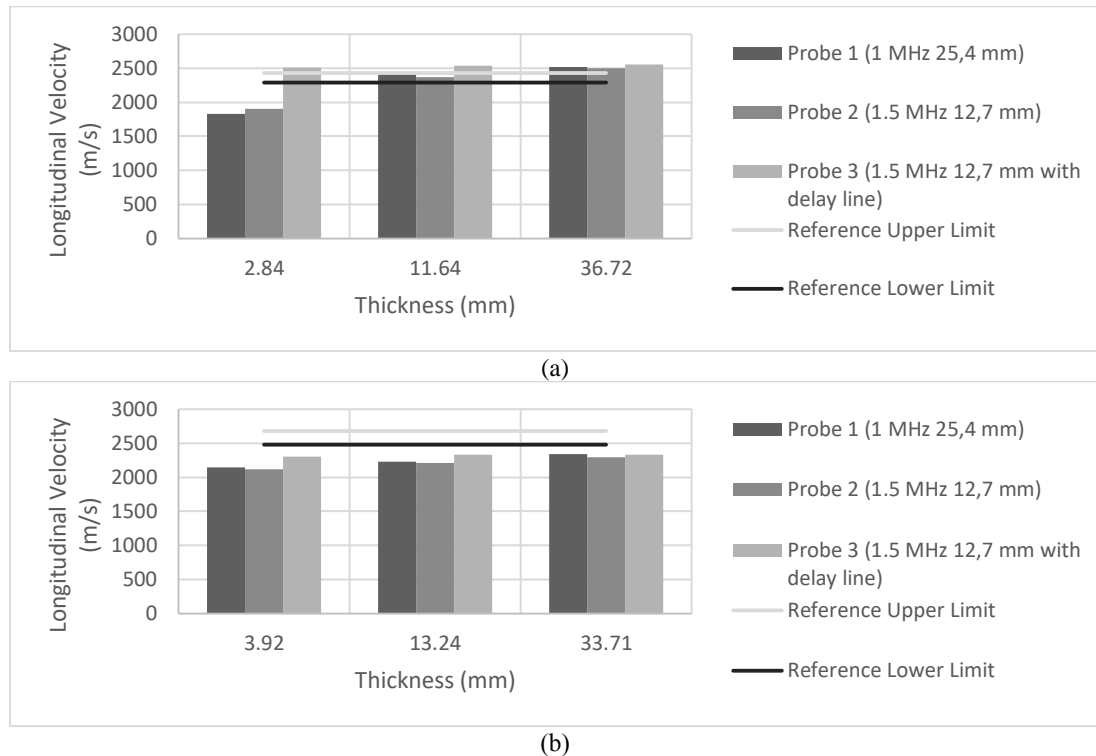


Figure 3.1 Longitudinal Velocity of (a) Polyester and (b) Epoxy from Reference and Ultrasonic Testing for Each Probe and Thickness

Longitudinal velocity measurements using probe 1 and probe 2 without delay line for specimens with thicknesses of 2.82–3.92 mm showed unfavorable results because they produced ultrasonic longitudinal velocity values that were much lower than in other thicknesses or measurements using probe 3 with delay line at the same thickness. Even so, measurements on specimens with thicknesses above 10 mm measuring using probe 1 and probe 2 without delay line have produced velocity values that approach the literature for polyester and epoxy.

The results of the ultrasonic longitudinal velocity measurements of each thermoset polymer using probe 3 with a delay line resulted in a relatively similar value for specimens with a thickness of 2.82 mm to 36.72 mm. Therefore, the use of probe 3 with delay line is considered the best compared to probe 1 or probe 2 which does not use delay line.

Table 3.3 exhibit the near field length on each probe and polymers. Probe 1 generates 60 – 66 mm near field length for polyester and 65 – 70 mm for epoxy. Probe 2 had lower near field length, 23 – 25 mm for polyester and 24 – 26 mm for epoxy. Probe 3 had lowest near field because of the subtraction of delay line thickness, 10 – 12 mm for polyester and 11 – 13 mm for epoxy. Compared to **Figure 3.1**, near field seems not affected longitudinal velocity measurement by ultrasonic testing in this experiment because there are not significantly differences between measurement results below and upper near field.

Table 3.3 Near field length

Polymers	Near field length (mm)		
	Probe 1 (1 MHz Ø 25,4mm)	Probe 2 (1,5 MHz Ø 12,7mm)	Probe 3 (1,5 MHz Ø 12,7mm dengan delay line)
Polyester	60 – 66*	23 – 25*	10 – 12**
Epoxy	65 – 70*	24 – 26*	11 – 13**

$$*N = \frac{D^2 f}{4V}, **N = \frac{D^2 f}{4V} - 13\text{mm (delay line)}$$

Besides near field, another phenomenon in ultrasonic testing is dead zone. The dead zone phenomenon will occur errors in ultrasonic reading for thin material so that it produces an incorrect longitudinal speed [10]. **Figure 3.2** shows dead zone displayed at ocilator screen.

**Figure 3.2** Dead zone displayed at ocilator screen

It is seen that the display area of the dead zone for this ultrasonic device is about 4 mm length. So that for specimens with a thickness less than the length of the dead zone, amplitude readings are not clearly visible when using probe 1 and probe 2 and causes errors in ultrasonic longitudinal velocity measurements. This is because on thin specimens, the entire thickness is the area of the probe to ringing down.

The effect of dead zone phenomenon will be lost when using probe 3 which has a delay line. This can be seen in the longitudinal velocity measurement with probe 3, in **Figure 3.1**, resulting in a more stable value (not too much different) even though there is a difference in specimen thickness.

3.3 Longitudinal Velocity Measurement Results

Table 3.4 shows the error of longitudinal velocity measurement of polyester and epoxy compared to literature, 4.18% for polyester and 6.29 for epoxy. **Table 3.4** also figures that Epoxy's apparent attenuation was 1.84 dB/mm, larger than polyester's 1.20 dB/mm.

Table 3.4 Longitudinal velocity, error, and attenuation on polymers and epoxy

Polymers	V _{Long} UT with ZIP (m/s)	Error of V _{Long} Measurement with ZIP (%)	Apparent Attenuation (α) (dB/mm)
Polyester	2532	4.18	1.20
Epoxy	2324	6.29	1.84

The larger error in longitudinal velocity measurement using ultrasonic testing commonly caused by larger apparent attenuation of materials [5]. Scattering, absorption and geometric are three parameters that effect the attenuation. Small discontinuities like boundaries between each crystal are the source of scattering [7]. In this case, there be influenced from density of materials, but the relationship of density and attenuation constant are inversely proportional as represent in **Table 3.1** and **Table 3.4**.

3.4 Young's Modulus Determination from Ultrasonic Testing

Table 3.5 figures that error of Young's modulus from ultrasonic testing compared to mechanical testing is 159% for polyester and 104% for epoxy. This large errors of calculation occurred even though errors of longitudinal velocity measurement with ultrasonic pulse echo testing with zero impedance probe were less than 7%, according to **Table 3.5**.

Table 3.5 Elastic Modulus from Ultrasonic and Mechanical Testing

Polymers	Young's Modulus UT (E_{UT}) (GPa)	Young's Modulus Tensile Testing (E_M) (GPa)	Error Elastic Modulus (%)
Polyester	4.62	1.78	159
Epoxy	3.36	1.65	104

Therefore **Table 3.6** shows that the result of polymer and epoxy Young's modulus calculation using longitudinal velocity from reference. This verify that **Equation 1** above is not applicable for polyester and epoxy used in this research and need to be modified.

Table 3.6 Elastic Modulus using Longitudinal Velocity References

Polymers	V_{Long} Reference (m/s)	Young's Modulus Calculation (GPa)	Young's Modulus Tensile Testing (E_M) (GPa)	Error Elastic Modulus (%)
Polyester	2290 [20]	3.78	1.78	112
	2430 [18]	4.25		138
Epoxy	2480 [20]	3.83	1.65	132
	2680 [18]	4.47		171

Table 4.6 proves that although using ultrasonic longitudinal velocities from the literature, the elastic modulus error is still very large, 112% -171%. This may be due to the formula that is used cannot be applied to thermoset polymers. Allegations related to this problem are the movement of thermoset polymer chains when exposed to dynamic loads on ultrasonic testing in contrast to chain movements when exposed to static loads on tensile testing [6]. This can occur because the variation of thermoset polymer movement is more diverse than metal. Variations in the movement of thermoset polymers are illustrated in Figure 4.11.

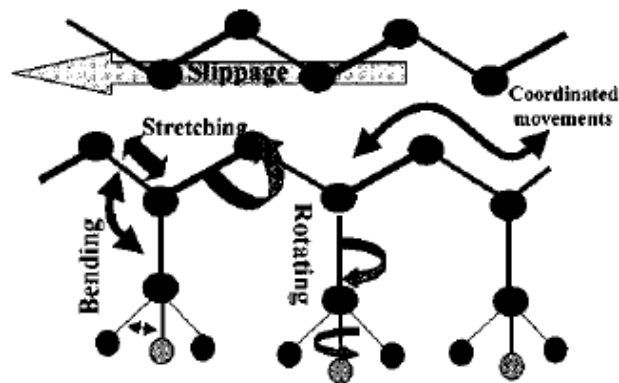


Figure 3.3 Crankshaft model to explain polymer chain movement [21]

Variations in the movement of the thermoset polymer chain make movement different for each dimension. Whereas in metals whose bonds consist of atoms, their movements tend to be the same in each direction in 3 dimensions, as illustrated in Figure 4.12 [3].

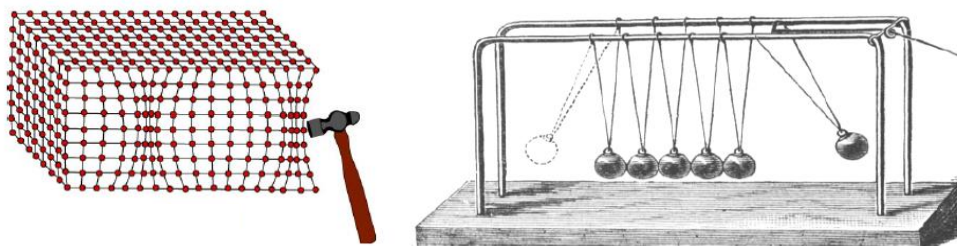


Figure 3.4 Atoms of metal movement illustration [3, 6]

So that standard equations can be used to determine the elastic modulus of thermoset polymers, then:

1. It is necessary to add the transversal direction velocity factor to the standard equation [6].
2. The standard equation requires other correction factors such as the nature of the thermoset polymer itself, for example density and attenuation [6].

4. Conclusions

The ultrasonic pulse echo method uses probe 3 with a diameter of 12.7mm, a frequency of 1.5MHz, and additional delay line is most suitable to measure the longitudinal velocity of polyester and epoxy with thickness of 2.82-36.72 mm. Probe 3 has a smaller diameter and additional delay lines thus eliminating the effect of dead zones. The elastic modulus error of thermoset polymer material through ultrasonic testing of pulse echo method compared to the mechanical modulus of elasticity is still very large, ie 102% for epoxy, and 159% for polyester. A large error is caused by attenuation and the use of the elasticity modulus equation (**Equation 1**) from ultrasonic testing that still requires modification.

5. Acknowledgements

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