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Elastic Modulus Determination of Thermoplastic Polymers with Pulse-Echo Method Ultrasonic Testing

Hermawan Judawisastra^{1*}, Claudia¹, Firmansyah Sasmita¹, Toni Agung P²

¹Materials Engineering, Faculty of Mechanical and Aerospace Engineering, *Institut Teknologi Bandung*, Bandung 40132, Indonesia

²Center for Material and Technical Product, the Ministry of Industry – Republic of Indonesia, Bandung 40132, Indonesia

* hermawan.judawisastra@material.itb.ac.id

Abstract. The determination of the elastic modulus of the structural thermoplastic polymers is especially required during in-service monitoring of the components. This elasticity modulus determination can simplify the inspection process and predict the lifetime of the polymer being used. The elastic modulus of the polymer can be determined by pulse-echo method testing ultrasonic which is easier to use than transmission method. This research aims to determine elastic modulus of thermoplastic polymers i.e. polymethylmethacrylate (acrylic), polyamide (nylon) and low density polyethylene (LDPE) calculated from measurement of velocity and attenuation of ultrasonic pulse-echo. The research begins by looking at the effect of the use of three different probes to ultrasonic velocity readings. Then elastic modulus of material polymers from ultrasonic velocity was calculated using standard equation. The elastic modulus was compared with the elastic modulus from mechanical testing. A linear model elastic modulus from ultrasonic reading which consists of velocity and apparent attenuation was developed. The results were elastic modulus value obtained from ultrasonic velocity and standard equation had a profound error 98% to 158%, especially for polymer with an eminently low density and high ratio of viscous property to elastic property. The attenuation from ultrasonic reading was influenced by the viscoelastic properties and the elastic modulus from ultrasonic velocity reading was influenced by the density. A linear model for thermoplastic elastic modulus determination based on ultrasonic pulse echo testing has been developed and could reduced the error to 3.45%.

Keywords: ultrasonic, pulse-echo, elastic modulus, polymer, attenuation, viscoelastic

1. Introduction

Thermoplastic polymer is commonly used as a structural material, but it can be degraded by exposure to temperature and relative humidity [1]. As a result the elastic modulus of polymers decreased than lead to a structural failure. By monitor its modulus and predict the lifetime of polymer component, failure can be prevented. Polymer also has a wide range of elastic modulus because of its microstructure variation, such as molecular weight distribution, branching, fillers and blends [2]. Therefore it is important to characterize even monitor the elastic modulus of polymer.

Tensile testing or destructive testing is the most familiar method to determine elastic modulus. There are several disadvantages of destructive testing for example it needs arduous sample



preparation and is impossible to use in the real time monitoring of in service structures. Meanwhile the field of non-destructive testing involves the identification without cutting apart or harming the object being tested. The most popular technique used is ultrasonic testing. It is able to use in the field and fast scan for repeatedly test. There are two approaches of ultrasonic: pulse echo and through transmission [3]. Elastic modulus and dynamic elastic modulus measurement with ultrasonic through-transmission method for polymers have been studied [4] and also wave velocity measurement in polymer with ultrasonic pulse-echo method has also been performed [5]. Pulse-echo is easier to use compare to transmission method and require only one side of the material. Both of these use high frequency sound waves on the order of 1-50 MHz [6].

The most commonly used indicators of material properties are velocity and attenuation or energy loss. The velocity depends on elastic modulus, poisson's ratio and density. The ultrasonic results are also influenced by the nearfield, diameter, and frequency of probe [7]. Recent research study is about energy loss discover a few additional characteristics of a material. Scattering, absorption and geometric are three parameters that effect the attenuation. Small discontinuities like boundaries between each crystal are the source of scattering [3]. In polymeric materials, especially thermoplastics, there may be influenced from density [8] and viscoelastic properties [9]. The lost energy is related to viscous properties used for chain disentangle and secondary bonds dissolve which cannot return to their original positions. Besides that, the amplitude readings from ultrasonic have not interrupted by nearfield phenomenon [7].

Therefore, this research aims to determine elastic modulus of thermoplastic polymers i.e. polymethylmethacrylate (PMMA or acrylic), polyamide (PA or nylon) and low density polyethylene (LDPE) calculated from measurement of velocity and attenuation of ultrasonic pulse-echo with regard to the effect of nearfield probe, density and viscoelastic polymer, and develop a linear model elastic modulus from ultrasonic reading which consists of velocity and apparent attenuation.

2. Materials and Methods

Thermoplastic polymers were as-received PMMA, PA, and LDPE polymers that were given cutting and grinding treatment to create a smooth surface. Material density was conducted with KERN ABJ-NM/ABS-N device that used archimedes principle at 25°C and ethanol 99.8% was used as the reference density.

Dynamic mechanical analysis test was conducted to obtain the viscoelastic properties of specimens that were storage modulus (E'), loss modulus (E'') and tan delta. The test itself was based on ASTM D 5023 [10] with 3 point bending type, 10 Newton load and 100Hz frequency at room temperature. Dimension of specimen was 50 x 20 x 2mm. Storage modulus (E') is a measure of elastic response of polymers. It measures the stored energy. Loss modulus (E'') is a measure of viscous response of polymers. It measures the energy dissipated as heat. Tan delta is the ratio of loss to the storage and called as damping. It related to how good a material will be at absorbing energy [11]. The value obtained from this test would be compared with the apparent attenuation and error elastic modulus from the ultrasonic test.

Tensile testing was conducted to obtain the elastic modulus of specimens. The test itself was based on ASTM D 638 [12] with speed of testing 5 mm/min and type I dimension. The value obtained from this test would be compared with the one from the ultrasonic test.

Ultrasonic test was conducted with GE USM 35X device with pulse-echo method to obtain the materials' longitudinal wave velocity. The probe used for the test were 1.5 MHz 12.7 mm diameter with delay line (probe 1), 1.5 MHz 12.7 mm diameter without delay line (probe 2), and 1 MHz 24 mm diameter (probe 3).

The nearfield zone of probe calculated with equation 1: where D is diameter probe (mm), f is frequency probe (Hz) and V is longitudinal wave velocity (mm/s). The nearfield of probe 1 and probe 2 is ± 20 mm and nearfield of probe 3 is ± 60 mm.

$$N = \frac{D^2 f}{4V} \quad (1)$$

Three times of longitudinal wave measurement in every section were conducted in every polymers and every probe. Measuring the velocity is based on ASTM E 494 [13] and apparent attenuation based on ASTM E 664 [14]. The sample code followed by materials- probe-sample thickness above (AN)/ below nearfield (BN). Table 1 showed the code of sample. The thickness samples of PMMA are 142, 40, 25, and 7 mm; PA are 98, 49, 25, and 16 mm; LDPE are 63, 30, 15, and 7 mm.

Table 1 Sample code (XX-XX-XX) of: materials – probe – sample thickness above/ below nearfield

	Thermoplastic Polymers		
	PMMA	PA	LDPE
Probe 1	PMMA-P1 -AN	PA-P1 -AN	LDPE-P1 -AN
	PMMA-P1 -BN	PA-P1 -BN	LDPE-P1 -BN
Probe 2	PMMA-P2 -AN	PA-P2 -AN	LDPE-P2 -AN
	PMMA-P2 -BN	PA-P2 -BN	LDPE-P2 -BN
Probe 3	PMMA-P3 -AN	PA-P3 -AN	LDPE-P3 -AN
	PMMA-P3 -BN	PA-P3 -BN	LDPE-P3 -BN

All of the longitudinal wave velocity obtained from the measurement would proceed to calculation process with equation 2: where E is elastic modulus, V_{long} is longitudinal wave velocity obtained from ultrasonic test, ρ is material's density, and ν is material's Poisson's ratio. For this experiment, Poisson's ratio that were used for calculations were literature's Poisson's ratio.

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

The apparent attenuation is determined by equation 3: where A_m and A_n are amplitudes of the m th and n th back reflection ($n > m$), and T specimen thickness.

$$\text{Apparent attenuation} = \frac{20 \log_{10} \frac{A_m}{A_n}}{2(n-m)T} \quad (3)$$

Linear regression was used to develop a model to find the best relationship between elastic modulus from mechanical testing as the dependent variable E_m and elastic modulus from ultrasonic testing E_{UT} and attenuation a as the independent variables. The correlation is calculated by Pearson method and parameter of linear equation is found by least square [15].

3. Results and Discussion

3.1 Physical, Mechanical and Viscoelastic Properties of Polymer

Table 2 showed the average of density, elastic modulus from tensile testing, viscoelastic properties which are storage modulus, loss modulus and tan delta, and Poisson's Ratio of three different types of thermoplastic polymers..

Table 2 Density, Elastic Modulus from Mechanical Testing, Viscoelastic and Poisson's Ratio of polymer

Polymers	Density (gr/cm ³)	Elastic Modulus from Tensile Test (GPa)	Viscoelastic Properties			Poisson's Ratio
			Storage Modulus (GPa)	Loss Modulus (GPa)	Tan Delta	
PMMA	1.19	1.99 ± 0.09	3.64	0.226	0.062	0.4 [16]
PA	0.95	1.06 ± 0.06	2.69	0.135	0.050	0.41 [17]
LDPE	0.89	0.29 ± 0.00	0.35	0.041	0.115	0.46 [17]

From the result, material polymers had a variation of density, elastic modulus, and viscoelastic properties and poisson's ratio. So that, these polymers could be used to find its influence in elastic modulus determination by ultrasonic testing.

The density of PMMA, PA and LDPE were 1.19 gr/cm³, 0.95 gr/cm³, 0.89 gr/cm³ respectively. The elastic modulus of PMMA, PA and LDPE were 1.99 GPa, 1.06 GPa, 0.29 GPa respectively. Density had a linear relation with an elastic modulus of materials. The denser materials would have a higher elastic modulus.

The storage modulus of PMMA, PA and LDPE were 3.64 GPa, 2.69 GPa, 0.35 GPa respectively. There was a linear relation between elastic modulus from tensile test and storage modulus from dynamic mechanical analysis. The higher elastic modulus from tensile test the higher storage modulus from dynamic test. But the storage modulus is not equal to elastic modulus because material response differently in quasi static tensile testing and dynamic testing bending [18].

The loss modulus of PMMA, PA and LDPE were 0.226 GPa, 0.135 GPa, 0.041 GPa respectively. The energy absorb use for chain disentangle and secondary bond dissolve. Tan delta or the ratio of viscous and elastic of PMMA, PA and LDPE were 0.062, 0.050 and 0.115. LDPE has the highest ratio of viscous and elastic, because the amorf and branched structure of LDPE make high loss of energy [19].

3.2 Evaluation of Ultrasonic Velocity Reading

Table 3 showed the velocity of several types of thermoplastic polymers from ultrasonic reading probe 1, probe 2, and probe 3 compared to velocity from reference [20]. Probe 1 diameter 12,7 mm with delay line showed more consistent velocity readings because velocity reading is not influenced by nearfield. Because the length of nearfield from ±20 mm decrease with the length of delay line 13 mm so the length of nearfield becomes only ±7 mm. Moreover, probe 1 is highly damped and low frequency delay line transducers. The low frequency characteristics make this probe good for penetrating thick or thin and highly attenuative materials [21].

For probe 1 the difference in speed between the material's thickness above the nearfield and below nearfield is 0.04% for PMMA, 0.3% for PA, and 5.4% for LDPE. Meanwhile probe 2 and probe 3 showed big differences velocity reading for the sample thickness which is below nearfield. The velocity difference for probe 2 is 10% for PMMA, 6% for PA, and 25% for LDPE and the velocity difference for probe 3 is 4% for PMMA, 5% for PA. A big difference

ultrasonic velocity in probe 2 and 3 compared to the velocity references was due to velocity reading with thickness below nearfield affected by nearfield. Nearfield is a region where wave interference phenomenon occurs, which will disrupt wave propagation and lead to misreading [22].

Table 3 Velocity of thermoplastic polymers from reference and Probe 1, Probe 2, Probe 3

Polymer s	V_{long} reference (m/s)	V_{long} ultrasonic (m/s)					
		Zip Probe 1 (Ø 12,7 mm with delay)		Probe 2 (Ø 12,7 mm without delay)		Probe 3 (Ø 24 mm)	
		N2 - AN	N2 - BN	N2 - AN	N2 - BN	N3 - AN	N3 - BN
PMMA	2690 [20]	2693	2684	2725	2443	2708	2604
PA	2400 [20]	2380	2379	2392	2243	2357	2230
LDPE	2237 [19]	2011	2126	2424	2984	-*	1856

*No sample thickness above 60mm

Therefore probe 1 diameter 12,7 mm 1,5 MHz with delay line are good for velocity reading for several thermoplastic polymer and several thickness. This probe will be used for calculation of apparent attenuation.

3.3 Comparison between Elastic Modulus from Mechanical Testing and Ultrasonic Testing

The results of elastic modulus from mechanical testing and elastic modulus from ultrasonic testing are shown in Table 4. Error of elastic modulus from ultrasonic E_{UT} is higher than elastic modulus from mechanical testing E_{M} . Error elastic modulus of PMMA is 98.5%, PA 119.5%, and LDPE 157.9%. Other research also showed that there are a profound error between elastic modulus from ultrasonic testing and elastic modulus from mechanical testing. Error can be caused by low density, attenuation and viscous elastic properties [8] [9].

Table 4 Elastic Modulus from ultrasonic testing and mechanical testing

Polymers	V_{long} Ultrasonic (V_{UT})(m/s)	Elastic Modulus UT (E_{UT}) (Gpa)	Elastic Modulus mechanical testing (E_{M}) (Mpa)	Error Elastic Modulus (%)
PMMA	2671	5.03	1.99	98.5
PA	2385	2.72	1.06	119.5
LDPE	1969	1.62	0.29	157.9

Figure 2 show the correlation between error elastic modulus calculation and density. The lighter polymer show the higher error elastic modulus, because the lower density polymer tends to have a bigger free volume and bigger viscous properties. Higher free volume makes the chain easier move between each other and did not return to their previous position. The displaced molecules have no tendency to slip back to their original positions, therefore these deformations are not recoverable absorb energy [23]. Meanwhile higher density of materials caused the waves propagate faster. This happened because there is less chain movements and less absorb energy [8]. Therefore the error elastic modulus in thermoplastic polymer is related to density.

The result from apparent attenuation ultrasonic testing and viscoelastic properties are shown in Figure 3. There is a strong correlation between attenuation from ultrasonic testing and viscoelastic properties. Attenuation is the combined effect of a number of parameters: absorption and scattering. Absorption is related to the viscoelastic properties, the more viscous polymers showed higher loss energy [24]. It is shown by tan delta, the ratio of loss

modulus and storage modulus of viscoelastic polymer. Higher tan delta means loss energy or viscous property is higher compare to storage modulus or elastic property. The energy absorb use for chain disentangle and secondary bond dissolve. Meanwhile scattering happens because of small reflection of material micro interfaces, such as crystalline and amorphous zone in polymer or bulky pendent group [7].

The highest error and attenuation is from LDPE followed by PA. The energy of ultrasonic wave excites the long range movement of LDPE and PA chains, so there is big absorb energy from LDPE and PA. The error would come from highly energy absorb by the microstructure of LDPE which is consists of branched and amorphous chain [24]. PA is less amorphous and branched chain than LDPE so it has less attenuation. Meanwhile PMMA has 92 % optical transmission [25] and it is denser so it is less scatter the energy than the other polymers.

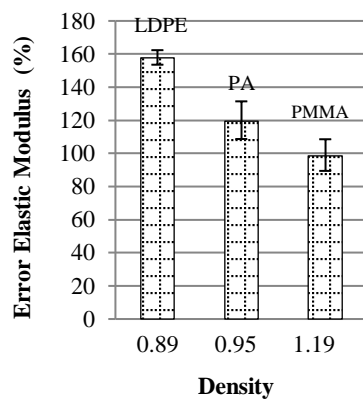


Figure 1 Error of elastic modulus and density

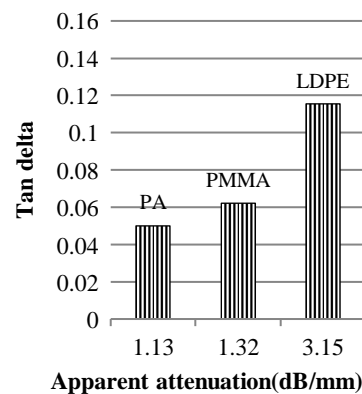


Figure 2 Viscoelastic properties and apparent attenuation

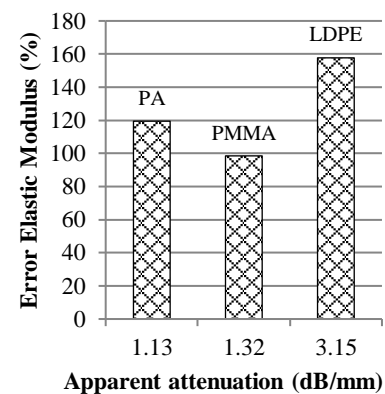


Figure 3 Error of elastic modulus and apparent attenuation

There is also a correlation nonlinear between apparent attenuation and error elastic modulus which is shown in Figure 4. The error value is greater if the apparent attenuation is greater. But there is a different trend in PA because attenuation is a combine effect of absorption and scattering [7].

Therefore the error of elastic modulus is still influenced by the viscoelastic and materials density. The viscoelastic properties have a strong correlation with the apparent attenuation.

3.4 Model of Thermoplastic Elastic Modulus Determination based on Ultrasonic Pulse Echo Testing

The correlation of modulus elastic from mechanical testing to modulus elastic from ultrasonic and attenuation is shown at Table 5. There were a strong linear correlation between elastic modulus from mechanical testing with elastic modulus and attenuation from ultrasonic testing. The model is shown by equation 4. Elastic modulus from ultrasonic testing affects elastic modulus from mechanical testing of 0.528. Attenuation affects elastic modulus from mechanical testing of 0.0128. While -0.161 is from constant parameters such as similar temperature [26], probe, and ultrasonic method [9].

Table 5 Correlation of modulus elastic from mechanical testing to modulus elastic from ultrasonic and attenuation

	Eut	a
Em	0.993	-0.771

$$\hat{E}_{\text{model}}(\text{GPa}) = 0.0128 \times a \left(\frac{dB}{mm} \right) + 0.528 \times E_{UT}(\text{GPa}) - 0.161 \quad (4)$$

Figure 5 shows comparison of elastic modulus from ultrasonic velocity calculated using standard equation (E_{UT}), elastic modulus from tensile testing (E_M), and elastic modulus from E_{UT} and attenuation from ultrasonic testing calculated with model (E_{model}). The use of developed model successfully reduced the error of elastic modulus from 98.5%- 157.9% to 2.16% -5.52% for 3 types of thermoplastic polymers.

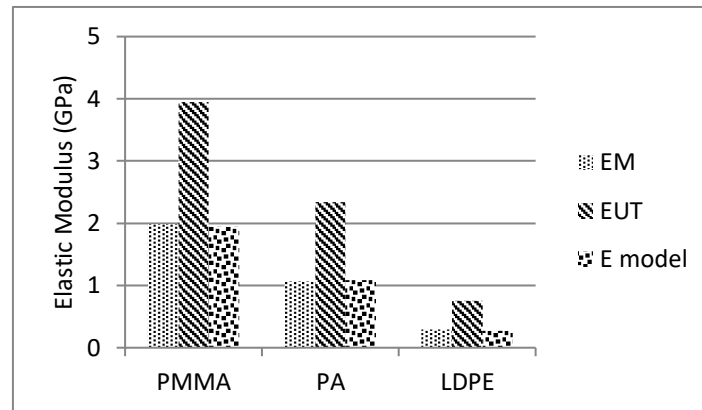


Figure 4 Comparison of elastic modulus of thermoplastic polymer from mechanical testing, ultrasonic, and model ultrasonic

3.4 Comparison between Elastic Modulus from Dynamic Mechanical Testing and Ultrasonic Testing

Table 6 Comparison between Elastic Modulus from Dynamic Mechanical Testing and Ultrasonic Testing

Polymers	Storage Modulus from DMA 3 point bending (GPa)	Elastic Modulus from Ultrasonik (E_{UT}) (Gpa)	Elastic Modulus from Tensile Testing (E_M) (Mpa)
PMMA	3.64	5.03	1.99
PA	2.69	2.72	1.06
LDPE	0.35	1.62	0.29

The difference between elastic modulus from ultrasonic E_{UT} and storage modulus from dynamic mechanical testing is smaller than elastic modulus from ultrasonic E_{UT} and elastic modulus from tensile testing. Elastic modulus from ultrasonic is near to the storage modulus from dynamic mechanical testing, because both ultrasonic testing and dynamic testing are describe dynamic mechanical behavior[4]. Ultrasonic wave is the particle making up the substance, and the restoring forces are the elastic bonds holding the substance together. While dynamic testing is sinusoidal load and response of material, so elastic and loss modulus can be determined. There was a slight difference between elastic modulus from ultrasonic and storage modulus from dynamic mechanical testing because the propagation of ultrasonic wave in polymers not only depend on elastic modulus, but also density and viscoelastic behaviors [4], which had been proved by experiment results in the previous section.

While the elastic modulus from 3 point bending was greater than elastic modulus from tensile testing because 3 point bending testing often results in a greater modulus of elasticity due to the smaller number of defects in the smaller test volume and there is a shear stress involvement [27].

4. Conclusions

The probe 1.5MHz 12.7 mm diameter with delay line or zero impedance probe (ZIP) results in more consistent reading for various thickness and enables the measurement of wave velocity and attenuation. The elastic modulus testing from ultrasonic on three types of thermoplastic polymers i.e. PMMA, PA and LDPE were still contained error from 98% up to 156%. Elastic modulus value obtained from ultrasonic testing from ultrasonic test had a profound error, especially for polymer with an eminently low density and high ratio of viscous property to elastic property. The attenuation from ultrasonic reading was influenced by the viscoelastic properties and the elastic modulus from ultrasonic velocity reading influenced by the density. A linear model for thermoplastic elastic modulus determination based on ultrasonic pulse echo testing has been developed and successfully reduced the error to 2.16 - 5.52% for three types of polymers.

5. References

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