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Study of Elastic Modulus Determination of Polymers with Ultrasonic Method

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***Abstract*— Elastic modulus is one of the mechanical properties of the material that often used in industry or research field as a benchmark to determine materials' performance in term of withstanding load without being deformed. Destructive testing is perpetually used to determine this property. However, destructive testing needs a sample, and in situ testing is implausible. Various types of materials are used in the production process of the industry, e.g., polymers. Polymers have time-dependent properties, which can result in high variety value. By principle, ultrasonic inspection, which depends on sound velocity and density of materials, can be used to determine elastic modulus. Ultrasonic test with through-transmission method has been studied to determine elastic modulus and dynamic elastic modulus for polymers. For the sake of quality control and engineering design, ultrasonic pulse-echo contact is preferable. Ultrasonic testing was conducted with GE USM 35X device, which is a Pulse-Echo method and also contact method type of ultrasonic testing machine. Experiments were conducted on several types of polymers with frequency and thickness as experiment parameters. With an input of specimens' thickness, materials' sound velocity (v) could be obtained. Thus results of v were counted to attain the elastic modulus. Comparison between ultrasonic testing results and mechanical testing results of polymers' elastic modulus were performed to analyze the data. In this research, elastic modulus value obtained from the ultrasonic test has a profound error, up to 65% (minimum) and 388% (maximum), especially for a polymer with an eminently low density. Further research should be conducted because of the attenuation effect. Also, lower probe frequency eases the detection of alternating ultrasonic wave. Specimens' thickness adjusted with near-field calculation can eliminate the near-field effect, which is a natural phenomenon of the ultrasonic wave. However, it would not have yielded an accurate value because an excessive thickness will give an attenuation effect.**

***Keywords*— ultrasonic; elastic modulus; polymers; attenuation; near field.**

I. INTRODUCTION

Elastic modulus is an important mechanical property of materials. This property measures the stiffness of materials or material's resistance to elastic deformation [1]. The most common test to determine elastic modulus is destructive testing, such as tensile and flexural testing. The testing is not only damage the product, but also high in expense, it is implausible to do an in-situ experience, and it is arduous to find some engineering constants for anisotropic materials [2]. Both in daily life and industrial practices, various kinds of materials are used. One of them is polymer [1], [3]. Polymers have some unique facets of material behavior need to be concerned. The most essential are time-dependent properties which can be affected by, such as, the stress level, operating temperature, and its structure (depend on, *e.g.* molecular weight, molecular orientation, and density) [4].

Ultrasonic testing is one of Non-Destructive Testing (NDT) method and commonly used to detect defects in materials [5]–[9]. The benefits of this method include flexibility, low cost, in-line operation, and supplying data in both signal and image formats for advanced analysis [10]. Nevertheless, with its principle, this method ought to measure materials' elastic modulus because mechanical wave behavior propagating in materials could be associated with materials' elastic properties. In general, the relationship between the velocity of sound in solids media and its density and elastic constants [11] is given by the following equation:

$$V = \sqrt{\frac{C}{\rho}} \qquad (1)$$

Where V is sound velocity, C is elastic constants, and ρ is density. This is further proven by standard of wave velocity measurement in isotropic materials and a formula that correlates wave velocity property of a material with its elastic modulus [12]. Previous study based on ultrasonic testing for determining elastic modulus of metals showed satisfying result. The experiment were conducted on AISI 304 stainless steel and aluminum with numerical error up to 3.1% and 2.1%, respectively [13]. The experiment confirmed that ultrasonic testing could be used to determine elastic modulus of metals. Another NDT method to determine dynamically elastic modulus is experimental modal analysis [14], [15]. Elastic modulus and dynamic elastic modulus measurement with ultrasonic through-transmission method for polymers have been studied [16], [17] and also wave velocity measurement in polymer with ultrasonic pulse-echo method has also been performed [2], [16], [18].

Due to so many appended factors on the behavior of polymers, properties such as elastic modulus excerpted as range value will be applicable as far as design in concerned, instead of as a means of quality control. To design a polymer component, it is crucial to get thorough information and data, at applicable service temperature, in terms of time-dependent behavior or viscoelastic of the polymers through the entire range of stress to be undergone by the component [4]. Furthermore, the ultrasonic pulse-echo method can resolve in connection with polymers' elastic modulus determination related to both quality control and engineering design. The ultrasonic pulse-echo is also relatively simple compared to the through-transmission method. However, research to determine elastic modulus with this method has not been conducted well despite the usual usage of the pulse-echo technique in the ultrasonic method. Therefore, this research emphasizes the effect of ultrasonic testing, specifically the pulse-echo method, and also of ultrasonic probe frequency in measuring the wave velocity of the polymer. The purposes of this research are determining the elastic modulus of polymers with the ultrasonic method, comparing the elastic modulus value obtained by the ultrasonic method and mechanical testing (which is based on ASTM D 638), and also observing the effect of ultrasonic probe frequency difference in elastic modulus determination.

II. Materials and Method

A. Material

Polymer specimens used in this experiment are grouped into two categories *i.e.* thermoplastic and thermosetting polymers. Each polymer group of specimens also divided into ultrasonic testing specimens, density test specimens, and tensile test specimens. Thermoplastic polymers that are being used were as-received PMMA, LDPE, and PA6 (Polyamide 6) polymers that were given cutting and grinding treatment to create a smooth surface. Thermosetting polymers that are being used were unsaturated polyester resin and epoxy resin that was made with resin and curing agent ratio according to industry standard. The thickness of the PMMA ultrasonic sample used for the experiment are 40.18 mm, 52.55 mm, and 59.91 mm, PA6 52.07 mm, 59.07 mm, and 79.01 mm, for LPDE two specimens with different thickness are needed which were 9.99 mm and 15.03 mm, polyester 45.73 mm, 66.82 mm, and 148.1 mm, and for epoxy 42.04 mm, 65.23 mm, and 120.1 mm. For density test, three samples for each polymer specimens were made with an average dimension of samples were 2.25 x 2.25 x 2.25 mm with error ± 0.25 mm, except for LDPE with 25 x 25 x 15 mm due to the initial dimension of specimens' limitation. Tensile test specimens were made based on ASTM D 638 [19].

B. Density Test

Density test was conducted with KERN ABJ-NM/ABS-N device that used the Archimedes principle. The density of test fluid was obtained based on device records which the researcher only needed to input the fluid test type and surrounding temperature. Test fluid used must have a density less than the sample has to make the sample submerged to the cantilever. Technical methanol was used for this test.

C. Mechanical Testing

Mechanical testing, namely the tensile test, was conducted to obtain the elastic modulus of specimens. The test itself was based on ASTM D 638 [19]. Tensile test machine to determine elastic modulus must be equipped with an extensometer. The value obtained from this test would be compared with the one from the ultrasonic test.

D. Ultrasonic Test

The ultrasonic test was conducted with GE USM 35X device with Pulse-Echo method to obtain the materials' longitudinal wave velocity. The frequencies used for the test were 1, 2 and 4 MHz, with probe diameter for 1 and 2 MHz probes were 24 mm and for the 4 MHz probe was 10 mm.

Fig. 1 the Ultrasonic equipment of GE USM35X series [20]

For this test, the thickness of the ultrasonic sample from two different sides of the same material and the peak appearing on the ultrasonic test device were the requirements to continue the test. Three times of longitudinal wave measurement in every section were conducted in every polymer and every probe frequency. Two methods are used for this ultrasonic test. Those are the 2-Point (2P) method, as described in GE Manual Book [21], [22], and Modified 2-Point (M2P) method as proposed in this study. The simplified procedure for the M2P method as schematically seen in figure 2 below. For polymers that happened to show more than one peak on display, the M2P method was used. The advantages of this method are only one side of the thickness of the material is needed, and after all the tests have been conducted, we conclude that this method has more stable results. But, if only one peak were shown on the display, 2P method was used. This method required us to input two different thickness of the same material (from the same sample will be preferred but from their difference side) to the machine and then calibrate it with the peak shown on display. Both of these methods and these processes after all could be conducted as those are this UT machine ability to calculate the longitudinal wave from all the data.

All of the longitudinal wave velocity obtained from the measurement would proceed to calculation process with equation 2:

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

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.

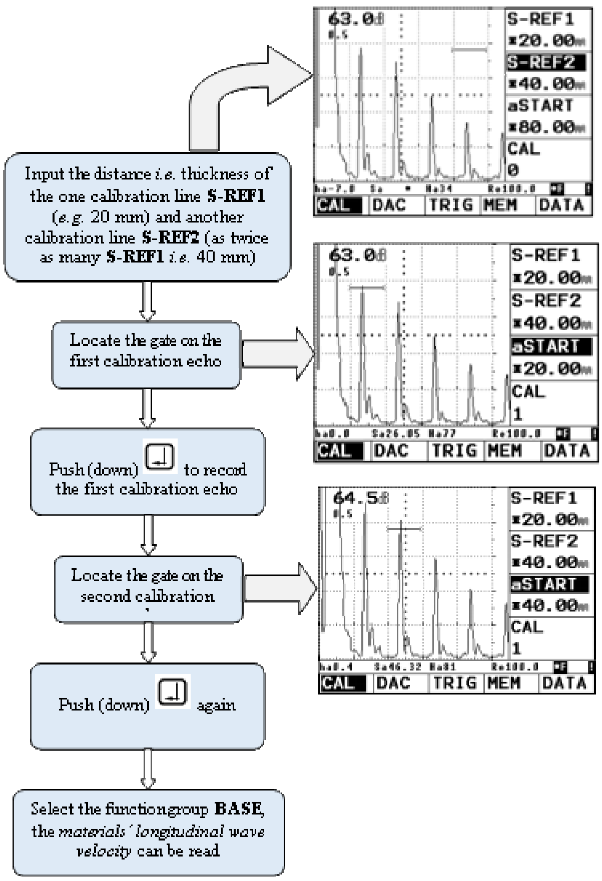


Fig. 2 Simplified M2P procedure on GE USM 35X to obtain materials' longitudinal velocity

For this experiment, Poisson's ratio that were used for calculations were literature's Poisson's ratio yet for the thermosetting polymers (Epoxy and Polyester), literature gave two elastic modulus results (maximum and minimum value) but for this paper, only the maximum value of Poisson's ratio will be used. The elastic modulus of wave velocity results from three times ultrasonic test was then determined. The average of these results would be calculated at the end.

III. Results and Discussion

A. *Elastic Modulus from Mechanical Test and Density Test Results*

Table 1 shows the elastic modulus from the mechanical test and density from five samples. The elastic modulus and density of PMMA were 3.2845 GPa and 1.1885 gr/cm^3. The elastic modulus and density of PA6 were 1.2017 GPa and 0.9546 gr/cm^3. The elastic modulus and density of LDPE were 0.26 GPa and 0.8965 gr/cm^3. The elastic modulus and density of Epoxy were 1.7115 GPa and 1.072 gr/cm^3. The elastic modulus and density of Polyester were 2.79425 GPa and 1.2028 gr/cm^3.

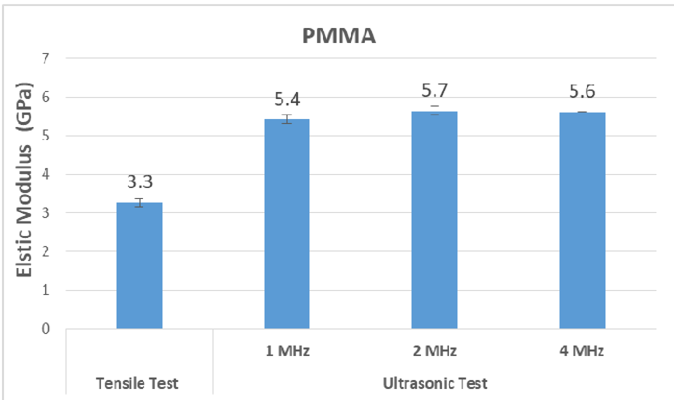
From the results, density has a linear relations with an elastic modulus of materials. The denser materials would have a higher elastic modulus. Even though polyester had a higher density than PMMA, but it has a lower elastic modulus. This might happened because defects, such as porosity, that exists more in thermosetting polymers than in thermoplastic polymers. Porosity in polymers will lessen the surface area of materials and acted as stress concentration spot.

TABLE I
Average of Elastic Modulus and Polymer Density from Mechanical Test Results and Density Test

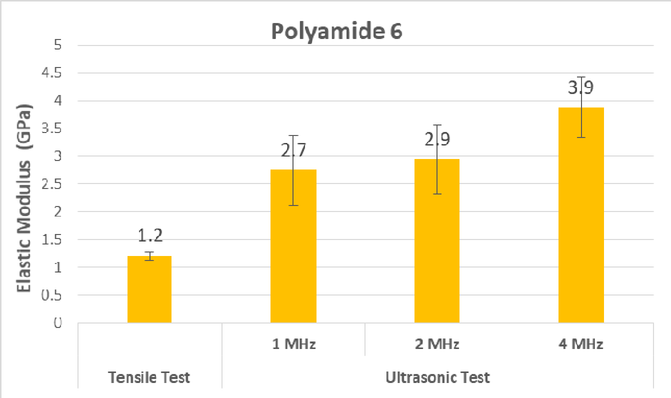
Polymer Specimen	Elastic Modulus (GPa)	Density Average (gr/cm^3)
PMMA	3.2845 ± 0.1049	1.1885 ± 0.0001
PA6	1.2037 ± 0.0711	0.9546 ± 0.0001
LDPE	0.260195 ± 0.0384	0.8965 ± 0.00166
Epoxy	1.7115 ± 0.2806	1.072 ± 0.0022
Polyester	2.79425 ± 0.0663	1.2028 ± 0.0022

B. *Comparison between Elastic Modulus from Mechanical Testing and Ultrasonic Testing*

(All these graphs below contained only the results from the M2P method (if appeared) as this method has more stable results. But if in the certain frequency, only one peak could be seen on the display, the result from 2P method are shown in the graphs)



(a)



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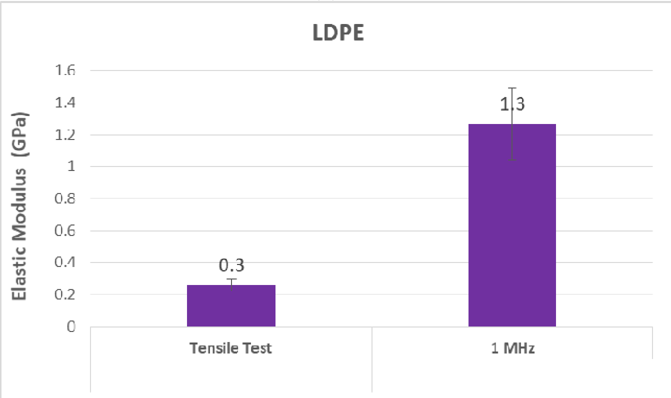
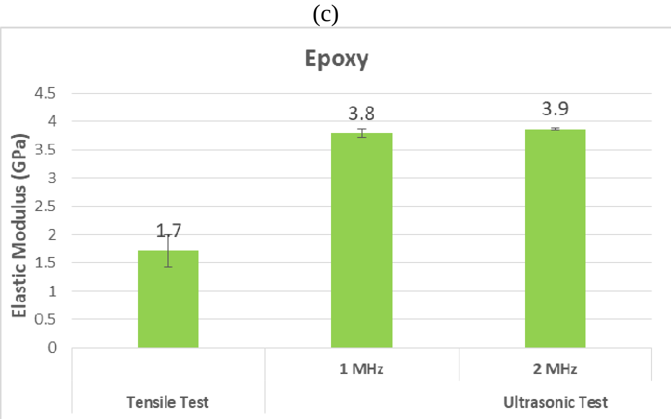


(c)



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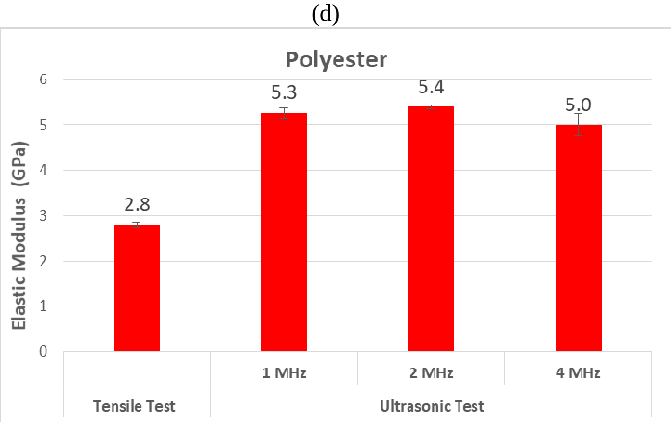

(e)

Fig. 3 Comparison between elastic modulus value from tensile test and ultrasonic test; (a) PMMA, (b) PA6, (c) LDPE, (d) Epoxy, (e) Polyester

Figure 3 shows the comparison between elastic modulus obtained from ultrasonic technique and mechanical test.

Elastic modulus error from ultrasonic test later calculated with formula below

$$Error = \left| \frac{E_{UT} - E_M}{E_M} \right| \times 100\% \qquad (3)$$

where E_{UT} is elastic modulus value from ultrasonic test and E_{UM} is elastic modulus value from mechanical test.

Table 2 shows the calculation of numerical error for Ultrasonic Testing relative to Mechanical Test.

TABLE II
ERROR OF ELASTIC MODULUS RESULTED FROM ULTRASONIC TESTING RELATIVE TO MECHANICAL TEST RESULT

	Error of Elastic Modulus Based on Ultrasonic Testing (%)		
Sample	**1 MHz**	**2 MHz**	**4 MHz**
PMMA	65.42	71.54	71.50
PA6	127.75	143.73	221.45
LDPE	387.49	-	-
Epoxy	121.39	125.59	-
Polyester	88.26	93.14	78.9

From all these tests, only PMMA and PA6 could show more than one peak for every frequency and for the higher frequency (4 MHz), it is only from lesser thickness, the peak could be detected. The rest of the polymers shows the same trend. For LDPE, it is only 2P method used because only in 1 MHz, we could see one peak. This owes to the fact that the higher the frequency, the lesser wavelength (λ) occurs then the ultrasonic wave propagating in material will be more sensitive towards defects and impurities. The low density of polymers also signify branched nature of polymer chains and high free volume. These two conditions increase the attenuation when wave propagates in material. As a result, only the low frequency could detect the returning ultrasonic wave and give an information about material's longitudinal wave velocity. The calculation of all the specimens' elastic modulus shows a huge error (minimum error is obtained from PMMA in 65.4% with 1 MHz frequency and maximum error is obtained from LDPE in 387.5% with 1 MHz frequency). Specifically for PMMA, previous study [18] also showed that there was good agreement in term of using 2 MHz probe frequency. The error between both studies was about 4.6%. Meanwhile, earlier investigation [14] showed that the result of dynamically elastic modulus for polyethylene had same trend as presented study in terms of comparison between mechanical testing and its non-destructive testing methods. Both studies show that the elastic modulus values are about three times larger than the values obtained from the mechanical testing. However, it is noticeable that there is a trend from all the materials that the higher the density, the higher the accuracy of elastic modulus (compared to the mechanical test result) as formulated in equation (1) above. The trend is explicitly also shown in figure 4 below in terms of 1 MHz frequency usage.

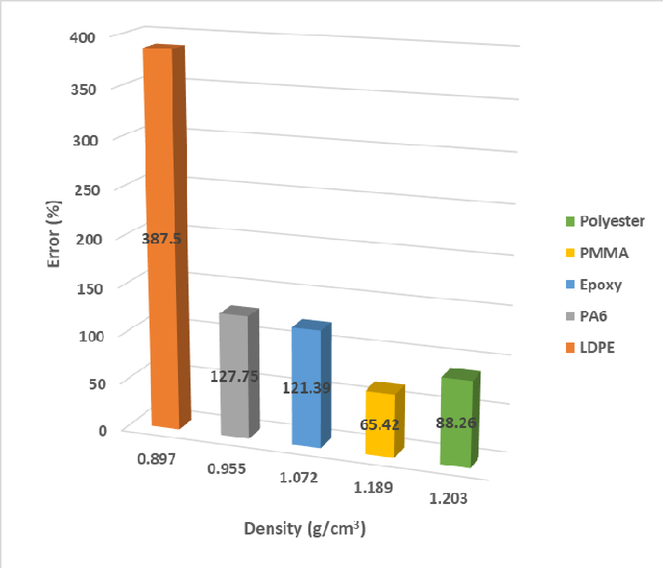

Fig. 4 Results comparison between error and density of polymers at 1 MHz frequency

But, elastic properties has more effect than density as we could see that in a material with bigger density, it tends to have an even higher E. From all the tests conducted, the tests with higher unstable longitudinal velocity and higher error percentage results came from the one with higher frequency and lesser thick specimens. This is the proof for a phenomenon that happened in ultrasonic, which is near-field [5]–[8]. Near-field is a region where wave interference occurs, which will disrupt wave propagation. Near-field in ultrasonic is calculated with formula below

$$N = \frac{D^2}{4\lambda} = \frac{D^2 \times f}{4V} \tag{4}$$

where N is *near-field* (m), D is probe/transducer diameter (m), λ is wavelength (m), f is frequency (Hz), and V is ultrasonic wave velocity in material (m/s), respectively.

TABLE III
CALCULATION OF NEAR FIELD OF TESTED POLYMERS

Sample	Frequency (MHz)	Near Field (mm)
PMMA	1	53.53
	2	107.06
	4	37.17
PA6	1	60
	2	120
	4	41.67
LDPE	1	69.23
Epoxy	1	56.69
	2	113.39
Polyester	1	62.88
	2	125.76
	4	43.67

From the ultrasonic test's elastic modulus result of the thermoplastic polymer, we could calculate that most of the specimens' thicknesses are in the near-field region (see table 3). It is caused by lack of the initial specimens' thickness so nearfield effect could be seen from the wave velocity measurement. However, this does not mean that near-field region could not be investigated since the ultrasonic wave still propagates in the material although wave that propagates in the near-field area would not be as stable as the one that propagates in far-field region. And for polymers, that has a natural slow longitudinal velocity properties, the near-field would be really high (compared to other materials like metals) and ultrasonic wavelength produced by the probe in the polymers would be shorter, as seen in equation 4 above. So, the sensitivity of the ultrasonic wave would be higher and increased sensitivity might distract ultrasonic wave even more to reach the other side of polymers (to measure the thickness of the polymers). Also from the research, we can conclude that far-field condition does not guarantee an accurate result because attenuation would still be increasing in an increasing thickness.

IV. CONCLUSION

Testing on five types of polymers were still contained error up to 65% (minimum) and 388% (maximum). Elastic modulus value obtained from ultrasonic testing from ultrasonic test had a profound error, especially for polymer with an eminently low density. Further research should be conducted because of the attenuation effect. From the test results, we can conclude that lower probe frequency used for testing enables ease alternating ultrasonic detection so it will allow the measurement of wave velocity. Specimens' thickness adjusted with near-field calculation can eliminate the near-field effect, which is a natural phenomenon of ultrasonic wave. However, it would not have yielded an accurate value because an excessive thickness will give an attenuation effect.

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