

MEASUREMENT OF ELASTIC PROPERTIES OF MATERIALS BY THE ULTRASONIC THROUGH-TRANSMISSION TECHNIQUE

MEDICIÓN DE LAS PROPIEDADES ELÁSTICAS DE MATERIALES POR EL MÉTODO DE TRANSMISIÓN ULTRASÓNICA

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ABSTRACT: The elastic mechanical behavior of elastic materials is modeled by a pair of independent constants (Young's modulus and Poisson's coefficient). A precise measurement for both constants is necessary in some applications, such as the quality control of mechanical elements and standard materials used for the calibration of some equipment. Ultrasonic techniques have been used because wave velocity depends on the elastic properties of the propagation medium. The ultrasonic test shows better repeatability and accuracy than the tensile and indentation test. In this work, the theoretical and experimental aspects related to the ultrasonic through-transmission technique for the characterization of elastic solids is presented. Furthermore, an amorphous material and some polycrystalline materials were tested. Results have shown an excellent repeatability and numerical errors that are less than 3% in high-purity samples.

KEYWORDS: ultrasound, nanoindentation, elastic properties

RESUMEN: El comportamiento mecánico de los materiales elásticos es modelado por medio de dos constantes independientes: el módulo de elasticidad y el coeficiente de Poisson. La medición precisa de estas constantes es indispensable en algunas aplicaciones, como son el control de calidad de elementos mecánicos y materiales patrones usados en la calibración de algunas máquinas de ensayos mecánicos. Dado que la velocidad de propagación de las ondas acústicas depende de las propiedades elásticas del medio donde se propagan, estas son usadas por la técnica de la transmisión ultrasónica, que es mucho más precisa y repetible que los métodos mecánicos de tracción e indentación. Este trabajo presenta los fundamentos teóricos y experimentales de la técnica de la transmisión ultrasónica, también muestra los resultados de ensayos realizados en varios materiales policristalinos y uno amorfo. Estos muestran excelente repetibilidad y errores numéricos menores al 3% para muestras de alta pureza de cobre y aluminio.

PALABRAS CLAVE: Ultrasonido, nanoindentación, constantes elásticas.

1. INTRODUCTION

The elastic properties of solids are key properties for the design of mechanically-loaded components [1]; especially, the elastic modulus and Poisson's ratio which are the properties that thoroughly describe the elastic behavior of isotropic materials. Literature reports elastic properties for many materials, such as non-pure metallic alloys, polymers, and ceramics. However, the values reported by different manufacturers are widespread due to the slight differences in the material properties. Another reason for variations in the reported values is the precision of the mechanical tests, which

depends on the quality of the experimental setup. Therefore, a reliable and reproducible technique is necessary when an accurate measurement is necessary, as it is, for example, in the quality control tests of some mechanical elements and standard materials used for the calibration of test equipment [2]. Ultrasound is a technique that meets all requirements mentioned above and which takes advantage of the principle that the propagation velocity of the waves through a solid medium depends on the solid elastic properties. This technique is so useful that it has become popularly used among other fields in composite materials [3], medicine, and biomaterials [4].

This paper describes the theoretical aspects of the through-transmission technique, showing the benefits and easiness of the implementation. It also describes an experimental setup that is simple but efficient. The elastic modulus and Poisson's ratio of aluminum and electrolytic copper of high purity, BK7 glass, and AISI 304 alloy were measured and the results were compared to the values found in the literature. Experimental results obtained by high-purity metallic samples show that the ultrasonic technique measures the elastic constants with an error percentage of less than 3%.

2. THEORETICAL ASPECTS

2.1 Fundamental aspects

Two types of bulk waves can be propagated in isotropic solids (longitudinal waves and shear waves). The longitudinal waves produce a displacement of material particles in the same direction of propagation. This displacement is therefore associated with normal stress. On the other hand, shear waves produce a displacement of the particles on a perpendicular plane to the direction of propagation. In the latter case, the displacements are associated with shear stresses as shown in reference 5 (see chap. 1, pp. 1-8). A first-order displacement of an infinitesimal size particle in a perfectly elastic material (which satisfies Hooke's law) is given by:

$$\tilde{n} \frac{\partial^2 u}{\partial t^2} = c \frac{\partial^2 u}{\partial x^2}, \quad (1)$$

Here, t is the time, u is the particle displacement, x is the propagation direction, ρ is the density of the unstressed solid, and c is a general elastic constant for the material.

For a harmonic wave, Eq. (1) has solutions for the form $u_x = u_0 e^{j(\omega t \pm Kx)}$, where K is called the propagation constant, and ω is the angular frequency.

For propagation in the longitudinal direction, the velocity of the wave (V_p) is:

$$V_p = \frac{\dot{u}}{K_p} = \sqrt{\frac{C_{11}}{\tilde{n}}}, \quad (2)$$

and the shear wave velocity (V_s) is:

$$V_s = \frac{\dot{u}}{K_s} = \sqrt{\frac{C_{11} - C_{12}}{\tilde{n}}}, \quad (3)$$

where C_{11} and C_{12} are independent constants of the stiffness matrix in simplified notation [6], K_p and K_s are the propagation constants for the longitudinal and shear waves, respectively. The stiffness constants are related to the elastic modulus (E), Poisson's ratio (ν), and the velocities of the waves as shown below [5]:

$$E = C_{11} - \frac{2C_{12}}{C_{11} - C_{12}} = \frac{V_p^2 - V_s^2}{(V_p/V_s)^2 - 1}, \quad (4)$$

$$\delta = \frac{C_{12}}{C_{11} - C_{12}} = \frac{V_p^2 - 2V_s^2}{V_p^2 - V_s^2}, \quad (5)$$

Generally, the shear stiffness of a material is less than its compression stiffness; therefore, the transverse wave velocity is smaller than the longitudinal ones. Generally, the experimental results show that the ratio V_p/V_s is approximately 2 for most metal alloys.

2.2 Wave velocity measurement

Figure 1 shows a schematic representation of a typical experimental setup used for measuring the elastic properties of a solid material. The technique uses two aligned piezoelectric transducers which are immersed in water. One works as a transmitter, and the other as a receiver. The sample with thickness d is placed between the transducers.

The velocities of the waves within the solid are obtained comparing the time elapsed for wave propagation in the cases with and without a sample.

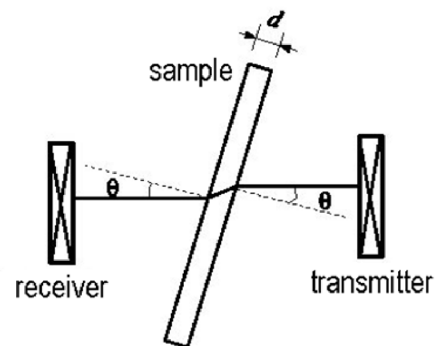


Figure 1. Schematic representation of the setup of the sample and transducers

When the wave reaches the water-solid interface with normal incidence, a portion of the longitudinal wave that is traveling in the water is reflected. Then, the remaining portion goes

through the sample and reaches the receiver. On the other hand, when the wave reaches the water-solid interface which has an oblique incidence, the wave succumbs to a phenomenon called *mode conversion*. During this conversion, part of the longitudinal wave becomes a shear wave. If the incidence angle is greater than a given value (called critical angle [θ_c]), then only a shear wave propagates through the solid. This shear wave is converted back into longitudinal one, when the wave reaches the opposite surface of the sample, i.e. the solid-water interfaces, and propagates into the water reaching the receiver. The value of the critical angle is governed by Snell's law. Thus, the mode conversion phenomenon makes the measurement of the propagation velocity of the two types of waves possible. In the work of Franco et al. [7] a more detailed study of the mode conversion phenomenon can be found.

Figure 2 shows the three typical echoes obtained during the ultrasonic test: When the solid sample is absent we get a longitudinal wave; when the sample is placed between the transducers with a normal angle $\theta=0$, we get another longitudinal wave; and finally, when the sample is rotated at an angle greater than the critical angle, i.e. $\theta>\theta_c$, a shear wave is obtained. When there is no sample in the water, the echo with the biggest amplitude is obtained. The delay between the longitudinal waves, in the cases with and without a sample, depends on the difference in the wave velocity in the sample and in the water. Consequently, a sample with a wave velocity similar to the wave velocity in the water is very difficult to characterize. The shear wave obtained for $\theta>\theta_c$ always has the greatest delay. The two additional waves shown in Fig. 2 are reflections of the longitudinal wave, which can be concluded because these echoes are repeated at a regular time period.

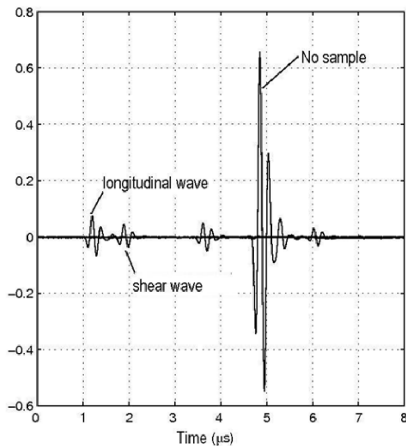


Figure 2. An example of the waves obtained during est.

By using the arrangement shown in Fig. 1, t_1 and t_2 are the transit time of the waves for both cases: normal and oblique incidence, and t_3 is the transit time when the sample is absent. The longitudinal wave velocity can be calculated by using the next equation [8]:

$$V_p = \frac{V_a}{1 + \frac{V_a(t_3 - t_1)}{\tilde{n}}}, \quad (6)$$

where V_a is the wave velocity in the water and d the thickness of the sample. On the other hand, for $\theta>\theta_c$, the equation for the shear wave velocity is [6]:

$$V_s = \frac{V_a}{\sqrt{\sin^2 \theta + \left[\frac{V_a(t_3 - t_2)}{\tilde{n}} + \cos \theta \right]^2}}, \quad (7)$$

where θ is the sample rotation. Equations 6 and 7 use the time difference between the waves, i.e. they use the delay between them ($\Delta t = t_3 - t_2$). For the materials tested here, the wave velocity through the sample is always greater than the velocity in the water. Therefore, the delays are always positive. However, in some materials, like some polymers, the wave velocity within the sample may be lower than the velocity in the water and, therefore, the delay has a negative sign. Special care must be taken when the delay time (Δt) is used instead of the individual time values, because a mistaken sign leads to a wrong velocity value. Furthermore, some materials may not have a critical angle or have a very large one. Consequently, the measurement of the shear wave velocity is not possible. In this case, taking a shear wave velocity equal to a half of the longitudinal one is a good approximation.

2.3. Time delay between two waves

There are several techniques for determining the delay time between two waves. The simplest one finds the maximum amplitudes of both waves and then calculates the time difference between those points. However, this technique may not be as accurate as is required. A better way to determine the delay in the time domain is calculating the envelope of the waves and determining the time of the maximum value of each envelope. This technique is the one most used in portable ultrasonic analysis devices.

The envelope of a signal can be calculated from the absolute value of the analytical signal. In an analytical signal, the negative frequency components of the Fourier transform (or Fourier *spectrum*) are discarded with no loss of information. The positive frequency is multiplied by 2 in order to maintain the amplitude.

Given a signal $x(t)$ with a Fourier transform $X(f)$, the following function contains only non-negative frequencies:

$$X_a(f) = X(f) \cdot 2U(f), \quad (8)$$

where $U(f)$ is the Heaviside step function. Calculating the inverse Fourier transform, the analytical signal $x_a(t)$ is obtained [9]:

$$x_a(t) = F^{-1}\{X_a(f)\} = x(t) + j \cdot \hat{x}(t), \quad (9)$$

This is a signal of complex numbers, where the real part is the original time signal and the complex part is its Hilbert transform [10]. Finally, taking the absolute value of the analytical function, the envelope of the signal is obtained:

$$E_x(t) = |x_a(t)|, \quad (10)$$

As an example, Fig. 3a shows a simulated signal composed by two symmetric echoes. Fig. 3b shows the envelope obtained from the absolute value of the analytical signal. It is clear that the delay can be obtained more easily from the envelope signal than from the original one. The following Matlab script example can be used to execute the procedure described above:

```
%----- Matlab -----
function dt12=delay(s1,s2,del1,del2,dt)
    H1=hilbert(y1);
    H2=hilbert(y2);
    [Hmax1 Imax1]=max(abs(H1));
    [Hmax2 Imax2]=max(abs(H2));
    dt12=(Imax2-Imax1)*dt+del2-del1;
%-----
```

In the script, s_1 and s_2 are the signal echoes bounded in appropriate temporal windows. del_1 and del_2 represent the time beginning of the respective temporal windows and dt is the frequency of the sampling of the signal. The Matlab command `hilbert` calculates the complete analytical signal.

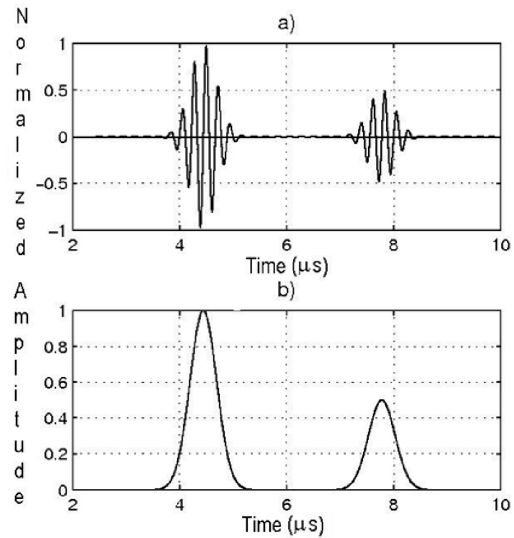


Figure 3. a) Simulated waves and b) the envelope obtained from the absolute value of the analytic function

When the wave propagates through long distances or propagates through materials of high attenuation, the wave form becomes distorted because the attenuation varies with the frequency. Therefore, it is more difficult to compare the waves in the time domain. Depending on the precision required, an analysis of the frequency domain is a better option, as suggested by Wu [11].

4. EXPERIMENTAL SETUP

In this work, four different materials were tested: high purity aluminum with a purity of 99.999%, electrolytic copper with a purity of 99.99%, BK7 glass (a standard borosilicate glass sample for nanoindentation equipments), and the stainless steel alloy AISI 304. The glass and stainless steel materials were chosen because they are widely used. Copper and aluminum of high purity were chosen because their elastic properties are well known, providing a good basis for comparison. An extensive study of the mechanical properties of these materials and also a comparison with other techniques can be found elsewhere [2,12]. All samples were flat specimens, cylindrical, or prismatic shaped, with an area between 9 and 16 cm² and a thickness between 2 and 3.2 mm. The geometry of the samples allows for their proper holding on the rotating base shown in Fig. 4b. The density of each sample was obtained dividing the weight by the volume. Table 1 shows the elastic properties reported in the literature (CES EduPack Software [13]) and the density obtained for the materials tested here.

Table 1. Physical properties of tested materials

Material	E (GPa)	ν	ρ (Kg/m ³)
aluminum	68	0.34	2686
electrolytic copper	117	0.35	8877
BK7 glass	70	0.21	2481
AISI 304 alloy	195	0.29	7940

The experimental setup, shown in Figure 4a, consists of an ultrasonic pulser/receiver (Panametrics 5072PR, Olympus NDT Inc, Waltham, MA, USA) and two commercial longitudinal transducer of 3.5 MHz each with a diameter of 12.7 mm (Panametrics V382, Olympus NDT Inc, Waltham, MA, USA). The signals were acquired with a sampling frequency of 500 MHz using an oscilloscope (Infinium 54820A, Agilent Technologies, Santa Clara, CA, USA) with a vertical resolution of 8 bits in averaged mode. Signals were processed using a Matlab script and, the rotation angle measurement was performed with a maximum error of $\pm 0.5^\circ$.

The measuring procedure is as follows: First, it is necessary to place the sample down correctly and to tie it down with the clamps. The sample is then rotated to find the angle at which the incidence of the wave is normal ($\theta=0$). That can be easily monitored using the oscilloscope because for normal incidence the delay of the wave is the smallest and the amplitude is the biggest. At this point, the first signal acquisition is done. Then, the sample is rotated above the critical angle. This angle can also be easily found with the oscilloscope because during the rotation of the sample, while the longitudinal wave gradually disappears, a new wave of smaller amplitude appears. The latter new wave, which is more delayed than the longitudinal one, is the shear wave. In order to achieve better accuracy, the sample must be rotated until the longitudinal wave disappears completely. The second signal acquisition is done. Finally, the sample is taken out from the water and the third acquisition is performed. The separation between the transducers must remain constant during the test. Moreover, in order to reduce error, large variations in the water temperature must be avoided because the velocity of the waves in solids and liquids is a function of temperature. In this work, the water temperature was maintained at around 22°C by using the air conditioning

system of the room. For each sample, the measurement procedure was repeated 6 times.

5. RESULTS AND DISCUSSION

Table 2 shows the measured velocities and elastic properties for all samples. Results are the mean and standard deviation of 6 repetitions. Velocities show a small standard deviation, with variations less than 2.9%. The elastic constants calculated from the measured velocities show a standard deviation of less than 4.4% and of 6.4% for the elastic modulus and the Poisson's ratio, respectively. These results show the repeatability of the measurement technique.

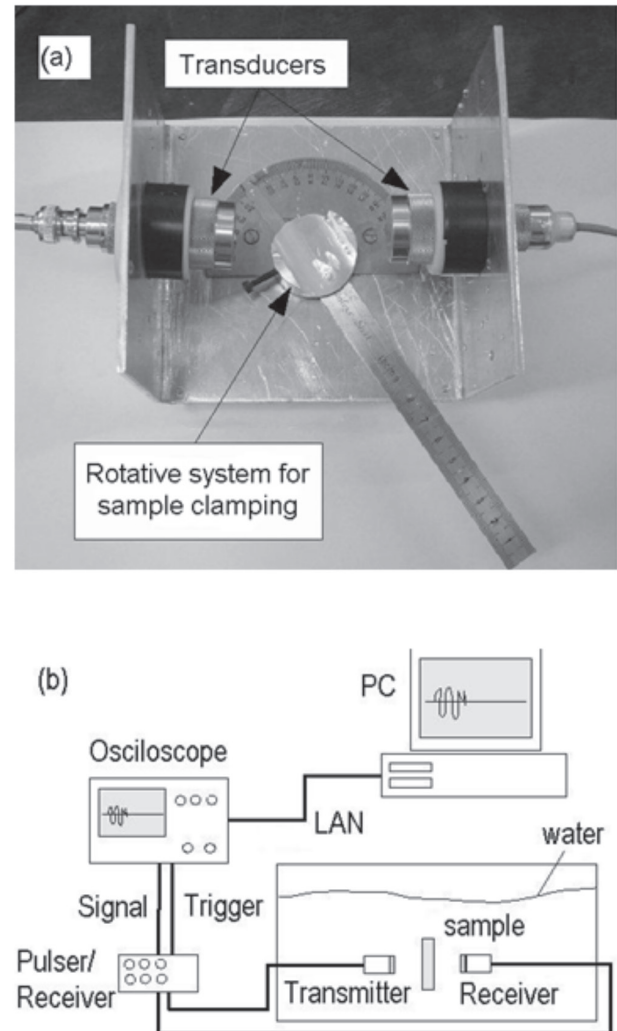


Figure 4. a) Sample clamping system and transducers b) Schematic representation of the experimental setup

Since the values of the elastic constants of pure samples (aluminum and electrolytic copper) are known with precision, they can be used to calculate the accuracy of the measured values. Table 3 shows the percentage of the relative errors between the elastic properties measured here (Table 2) and those reported in the literature (Table 1). In the case of the aluminum and copper samples, the biggest error was 2.3% for the elastic modulus and 2.1% for Poisson's ratio. The values of the elastic properties reported in the literature for these pure materials are unique. Therefore, it is possible to conclude that the ultrasonic results are quite accurate and reproducible.

For all samples the percentage error increases up to 3.6%. However, the value of the elastic constants for AISI 304 alloy and BK7 glass varies slightly in the literature, and to establish a precise error value is difficult. The error occurs because the chemical composition of the glass and steel samples varies from one manufacturer to another.

The numerical value of Poisson's ratio exhibits a high sensitivity to the variations of the longitudinal and shear velocities. Therefore, a small error in the velocity measurement drastically affects Poisson's ratio. That can be useful for the analysis of the obtained results. In this case, an obtained Poisson's ratio which is different from the value reported in the literature may reveal a measurement problem.

Table 2. Results for E and ν

Material	V _p (m/s)	V _s (m/s)	E (GPa)	ν
aluminum	6357 ± 19	3073 ± 5	68.4 ± 0.2	0.347 ± 0.002
electrolytic copper	4808 ± 62	2230 ± 44	119.7 ± 4.0	0.355 ± 0.008
BK7 glass	5798 ± 44	3444 ± 15	72.2 ± 0.7	0.227 ± 0.005
AISI 304 alloy	5646 ± 13	3019 ± 89	188.2 ± 8.3	0.299 ± 0.019

Table 3. Relative error between elastic properties reported in the literature (subindex "l") and those measured by ultrasound technique (subindex "u")

Material		$\frac{ \tilde{\sigma}_l - \tilde{\sigma}_u }{\tilde{\sigma}_l} \cdot 100$
aluminum	0.6	2.1
electrolytic copper	2.3	1.4

BK7 glass	3.1	3.1
AISI 304 alloy	3.6	3.1

Another aspect which is important to analyze is the error due to the measurement of the rotation angle of the sample. A hypothetical material with $V_p=6000$, $V_s=3000$ m/s, and $\rho=3000$ Kg/m³, which corresponds to an elastic modulus of 72 GPa and a Poisson's ratio of 0.333 is analyzed. In the experimental setup, the maximum uncertainty in the rotation angle measurement is $\pm 0.5^\circ$, and the typical critical angles for our samples are less than 20° . In these conditions, the maximum calculated error in both mechanical properties is about 3%.

One of the most important advantages of the trough transmission technique is that most of the necessary equipment (an oscilloscope and a computer) are commonly found in many engineering laboratories. Although, the pulse/receiver and the transducers are specifically designed for ultrasonic applications, they are provided by several companies that supply non-destructive testing equipment at relatively cheap prices.

6. CONCLUSIONS

The ultrasonic through-transmission technique is versatile and precise in determining the elastic properties of isotropic materials. The characterization of pure samples of copper and aluminum showed that the method has an accuracy which is better than 2.3% for these metallic materials using the experimental setup used in this work.

As Poisson's ratio is highly sensitive to inaccuracies in the measurement of the wave velocities, the measured ratio can be compared to values in the literature in order to test the validity of the results.

The error induced in the measurement of the elastic properties due to uncertainty in measuring the rotation angle of $\pm 0.5^\circ$ is less than 3%. However, we recommend implementing a more accurate system than the one used in this work.

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