Direct measurement of ultrasonic velocity of thin elastic layers

Mingxi Wan, Bei Jiang, and Wenwu Cao
Intercollege Materials Research Laboratory, The Pennsylvania State University, University Park, Pennsylvania 16802

(Received 2 May 1996; accepted for publication 24 August 1996)

This letter reports a simple new technique for the ultrasonic velocity measurement on thin elastic layers of fractional wavelength thickness using broadband transducers. An external trigger to the oscilloscope with continuously variable frequency and an intensity enhancing strobe signal allow direct measurement of the wavefront time shift down to 13 ns. The technique makes it possible to measure the sound velocity of thin layers with the thickness down to $10^{-2}\lambda$. Using a pair of 2.25-MHz transducers, we have accurately measured the sound velocity of a 26-μm-thick aluminum foil. © *1997 Acoustical Society of America.* [S0001-4966(97)04112-X]

PACS numbers: 43.58.Dj, 43.20.Jr, 43.35.Zc, 43.35.Cg [SLE]

INTRODUCTION

There are many situations of technological importance in which one wishes to carry out the elastic property characterization of thin layer materials; for example, bonding layers in structural materials, layers in composite structures, and thin film materials on thick substrate. Over the past 30 years, a wide variety of ultrasonic techniques have been reported for the measurements of wave velocities or the thickness of a thin single elastic layer. Among all classical methods are pulse echo,[1] resonance testing,[2] pulse interference methods,[3] and laser ultrasonic method.[4] These traditional techniques measure the time delay in well-separated echoes. However, as the layer thickness decreases, the time interval between two successive echoes from the front and back surfaces of the specimen decreases. Eventually, all of the classical methods fail when the echoes become inseparable.

In many applications, the thickness of the elastic layer, h, is of the order of 10–100 μm, and may even be imbedded inside a structure or blocked by another object. If using the well-separated pulse method to characterize such thin layers, the frequency would have to be larger than 150 MHz. The use of such a high frequency not only drastically increases the cost, but also limits the application to low loss materials because of the very short penetration depth of high-frequency waves.

Several years ago, a frequency domain method[5] for ultrasonic nondestructive evaluation of thin specimens was developed and improved by means of combining the standard FFT methods with conventional ultrasonic method. This method is based on the fact that a thin specimen in the time domain takes the form of a thick specimen in the frequency domain. A time domain method for the measurement of ultrathin specimen was also developed in 1993 in which low-frequency ultrasonic wave was also used.[6] However, for these frequency and time domain methods, digital oscilloscopes with a very high sampling rate and specialized data analyses are required. Sub-half-wavelength thickness thin layers can be measured using these methods down to $10^{-1}\lambda$.

In this letter we describe a new approach for the measurement of a thin elastic layer with the thickness in the order of $10^{-2}\lambda$. The idea is to manipulate the triggering rate and the strobe sweeping to fully utilize the maximum sensitivity of the oscilloscope. We call the technique variable trigger and strobe (VTS) method. Using this technique, the time shift caused by placing and removing the sample from the acoustic pathway between transmitting and receiving transducers can be accurately measured down to the maximum capacity of the oscilloscope (0.01 ns for the one available to us). Since only the starting point of the transmitted wave is used, a low-frequency transducer can be used to measure these samples. This gives us the advantage of deep penetration and the ability to measure thin layers that are sandwiched in lossy materials.

I. THE VTS METHOD AND EXPERIMENTAL PROCEDURE

A schematic of the experimental apparatus is shown in Fig. 1. A pair of accurately matched broadband water immersion longitudinal wave transducers with a center frequency of 2.25 MHz were used for generating and receiving the ultrasonic waves. The distance between the two transducers is adjusted to be equal to twice the focal length, and the specimen is placed in the focal region of the transducers. The incident wave travels in the direction normal to the specimen. For a single-layer specimen, the displayed pulse signal from the time t_0, at which the received signal appears, to the time t_0+2h/c will not have the effect of multireflection interference. Therefore, we can get the longitudinal velocity c by measuring the time shift Δt of the starting point of the transmitted waves caused by including the specimen in the acoustic pathway,

$$c=\frac{c_w h}{h-c_w \Delta t}, \tag{1}$$

where h is the sample thickness and c_w is the sound velocity of water (reference medium). The time shift Δt can be either positive or negative depending on if the c is faster or slower than c_w.

Because the technique is to compare the travel time with and without the sample in the acoustic pathway, it is insensitive to other objects in the pathway since their contributions can be included in the travel time without the sample.

0001-4966/97/101(1)/626/3/$10.00

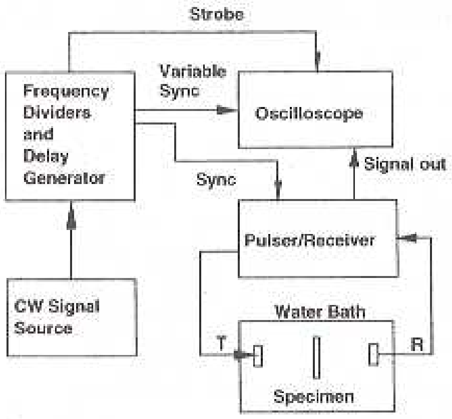

FIG. 1. Schematic diagram of the experimental setup.

The only difference between having water as the base medium or including other objects in the base medium is the intensity level. Since the technique allows the use of relatively low frequencies, it is possible to penetrate even lossy materials in the pathway. This characteristic makes the technique very useful for characterizing a thin layer in a sandwich structure, such as the bonding layer.

Although most oscilloscopes have subnanosecond resolution in the horizontal scale, it is not possible to use the highest resolution in most cases because of the limited display of time interval on the display window. Most of the analog oscilloscopes, such as the one used in this experiment, do not have time delay function; therefore, it is impossible to follow the wavefront when changing the horizontal resolution. One way to utilize higher resolution of the oscilloscope is to use higher external triggering frequency; however, it will mix up the received pulse signals, making it impossible to distinguish different echoes. Considering the analog oscilloscope has an intensity regulating input, the selected echo is highlighted by using a strobe signal from a Matec 122B decade dividers and delay generator. By reducing the background intensity, this selected signal can be singled out for measurement using the high resolution scale of the oscilloscope with high external triggering frequency.

General procedure for the VTS method: As shown in Fig. 1, the Matec 110-CW signal source is integrated with a highly stable oscillator tunable over a range of approximately from 11 to 52 MHz. The frequency output of the oscillator is divided by 100, and then is further divided by 100 in the Matec 122B decade dividers and delay generator. A more stable pulse signal with a frequency range of 1.1–5.2 kHz is obtained. This frequency is then used to trigger the JSR DPR 35 pulser/receiver and the Tektronix 2465 oscilloscope. The received pulse signals are sent from the DPR 35 to the oscilloscope for display. The strobe from the Matec 122B is coupled to the Z axis (intensity) of the oscilloscope and the front portion of the first transmitted wave signal is brightened by adjusting the strobe delay, width, and amplitude. The intensity of the oscilloscope must be carefully adjusted together with the strobe amplitude to obtain a proper display of the front portion of the transmitted wave signal so that the unwanted portion of the wave signal and the background sweep line will disappear. The front portion of the transmitted wave is then expanded horizontally to the proper resolution of the oscilloscope according to the level of time shift to be expected. The signal will be out of the screen from the right-hand side as the resolution of the oscilloscope increases. Now the external trigger is switched from the Matec 110 signal source to another scale which has 100 times higher triggering frequency. The brightened front portion of the first transmitted wave (or wave train) will reappear on the screen. By adjusting the frequency of the signal source to change the triggering frequency of the whole system, we can place the starting point of the front portion of the first transmitted wave anywhere on the screen. As a rule of thumb, place the signal on the left-hand side of the screen if the wave speed in the specimen is faster than that in water, and to the right-hand side if the compressional wave velocity in the sample is slower than that in water. After the thin layer sample is taken out of the ultrasonic field, the starting point of the front portion of the first transmitted wave shifts to a new position. This shift can be directly measured by using the scale bars on the screen of the scope. Then the compressional velocity in the thin layer of known thickness can be calculated using Eq. (1). Vice versa, if the velocity is known, the time shift can be used to measure the sample thickness.

II. RESULTS AND DISCUSSIONS

The main objective of this work is to develop a simple technique suitable for the characterization of ultrathin elastic layers of only a fraction of a wavelength in thickness. We

TABLE I. Wave speed measurement data for single elastic layers.

Specimen No.	Material	Thickness (mm) ± 3 μm	Time shift (ns)	Velocity (mm/μs)	h/λ	Sweep speed (ns/Div.)
1	Glass	1.247	613.4	5.579	0.503	100
2	PZT	0.236	91.70	3.539	0.150	20
3	Transparency #1	0.104	24.55	2.298	0.102	20
4	Transparency #2	0.096	18.45	2.088	0.103	20
5	Plastic shopping bag	0.058	16.55	2.592	0.050	10
6	Aluminum foil	0.026	13.26	6.206	0.009	5

TABLE II. Wave speed measurement data for elastic thin layers on substrates.

Specimen No.	Coating material	Thickness (mm) ±3 μm	Time shift (ns)	Velocity (mm/μs)	h/λ	Sweep speed (ns/Div.)
3	Transparency #1	0.104	25.00	2.321	0.101	20
4	Transparency #2	0.096	19.70	2.146	0.101	20

have measured a number of specimens using the newly developed VTS method. The results for a single layer and for a thin layer on a thick substrate are presented in Tables I and II, respectively. All results were obtained under constant temperature of 20 °C; thus the value of c_w is taken as 1.49 mm/μs. The substrate used for testing the coating specimen are glass plates of 1.247 mm in thickness.

As shown in Tables I and II, the measurements were performed on several materials with thickness ranging from 0.026 mm to 1.24 mm. The central frequency of the broadband transducer used in the experiments is 2.25 MHz, hence, the thickness range corresponds to 0.009 λ–0.503 λ. From a systematic error analysis (to be reported later), we found that the total error in the velocity measurement is less than 3%. Since most of the materials measured do not have known velocity values, we can only compare the longitudinal velocity of the aluminum foil which has been measured in bulk form.[7] Our measured value of 6.206 mm/μs is 3% smaller than the bulk value of 6.400 mm/μs. Since this difference is within our experimental accuracy, we cannot attribute it to the difference between bulk and foil materials. Generally speaking, the VTS method works better for high sound velocity materials which will lead to larger time shift, as shown in Table I. In other words, it is more sensitive for metal, ceramic, and glass than for plastic and polymer products.

As the thickness of the thin layer or the velocity difference between the sample and water decreases, we must increase the horizontal magnification on the scope. For the ultrathin aluminum foil (specimen #6 in Table I) with $h/\lambda = 0.009$, the vertical scale also must be enlarged in order for the starting point to be located more accurately on the screen.

The signal source and decade dividers used are very stable; no drifting of the signal was observed. However, water disturbance caused by removing the specimen out of the ultrasonic field does cause the signal to oscillate. Therefore, it is necessary to remove the sample slowly and to wait a few minutes before taking the comparison data.

For the ultrathin layers with $h/\lambda < 10^{-2}$, the noise in the receiver output could affect the accuracy. Especially when the vertical amplitude has to be enlarged enough on the screen together with time axis expansion, the ambiguity of the starting point resulting from noise is the main error source of wave velocity measurement for ultrathin layers of $h/\lambda < 10^{-2}$ using this VTS technique. It is expected that the VTS technique could be further improved by using digitizing oscilloscope combined with numerical signal average techniques. This technique could be used in reflection mode if the coupling between the transducer and the sample is accurately controlled.

ACKNOWLEDGMENT

This work is supported by the Office of Naval Research under Contract No. N00014-93-1-0340.

[1] E. P. Papadakis, "Ultrasonic velocity and attenuation: Measurement methods with scientific and industrial applications," in *Physical Acoustics Principles and Methods*, edited by W. P. Mason and R. N. Thurston (Academic, New York, 1976), Vol. XII, pp. 277–374.

[2] D. I. Bolef and M. Menes, "Measurement of elastic constants of RbBr, RbI, CsBr, and CsI by an ultrasonic CW resonance technique," J. Appl. Phys. **31**, 1010–1017 (1960).

[3] H. L. Meskimmin, "Pulse superposition method for measuring the velocity of sound in solids," J. Acoust. Soc. Am. **33**, 12–16 (1961).

[4] A. C. Tam and H. Coufal, "Photoacoustic generation and detection of 10-ns acoustic pulse in solids," Appl. Phys. Lett. **42**, 33–35 (1983).

[5] V. K. Kinra and V. Dayal, "A new technique for ultrasonic-nondestructive evaluation of thin specimen," J. Exp. Mech. **28**(3), 288–297 (1988).

[6] V. K. Kinra and C. Zhu, "Time-domain ultrasonic NDE of the wave velocity of a sub-half-wavelength elastic layer," J. Test. Eval. **21**(1), 29–35 (1993).

[7] W. P. Mason, "Acoustic Properties of Solids," in *American Institute of Physics Handbook* (McGraw-Hill, New York, 1972), 3rd ed.