Improved Ultrasonic Spectroscopy Methods for Characterization of Dispersive Materials

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***Abstract*—Transmission ultrasonic spectroscopy method has been successfully implemented for the characterization of piezoceramics at high frequencies. There are, however, still some intrinsic error sources that limit the accuracy of the method. In this paper, two improved ultrasonic spectroscopy methods are presented, which can reduce the number of pre-required parameters and reduce another error source. The two improved methods were used to measure the frequency dispersion of phase velocity and attenuation of doped piezoceramic lead zirconate titanate (PZT-5A); results were compared with those obtained from the conventional method. The advantages and limitations of each method are discussed.**

I. Introduction

Broadband ultrasonic transducers with center frequency over 30 MHz have become more and more popular in medical ultrasonic imaging field (ophthalmology, dermatology, or intravascular examinations). Design of such high frequency transducers requires a complete knowledge of material properties at high frequencies. The dispersions of velocity and attenuation of most transducer materials are not known for frequencies over 30 MHz. After Sachse and Pao [1] developed the through-transmission ultrasonic spectroscopy to determine the dispersion relation in dispersive solids in 1978, this technique has been the main tool to characterize solid materials [2]–[7]. Recently, we have successfully applied this method to characterize piezoelectric ceramics at frequencies up to 65 MHz [8]. Using this method, two ultrasonic pulse signals, one without and one with the specimen inserted in between two broadband transducers, are recorded separately. The frequency dependence of phase velocity and attenuation can be determined from the phase and amplitude spectra of these two pulses when the speed of sound in water and the thickness of the specimen are known. The error in thickness measurement using micrometer and the accuracy of the water velocity used will unavoidably affect the accuracy of the measurements. It was shown that the water velocity could be eliminated if both reflection and transmission signals are used at the same time [9]. However, we found that this technique is not adequate for high frequency measurements because the reflection signal has 180°phase shift so that the reflected signal can be partially canceled by the electric leaking signal, particularly for thin samples, introducing additional uncertainties.

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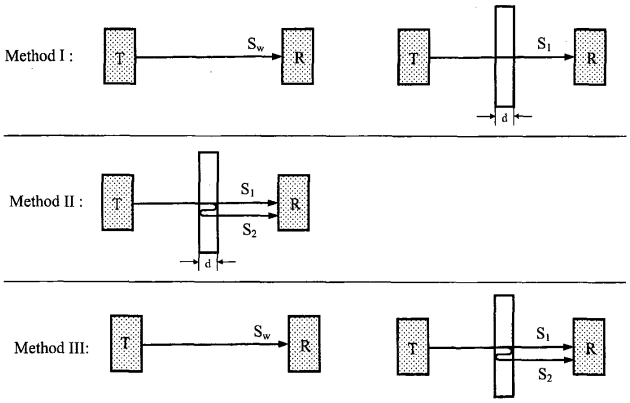

Fig. 1. Signal paths in the conventional and two improved through-transmission ultrasonic spectroscopy methods.

In this paper, we report an investigation on two other improved through-transmission ultrasonic spectroscopy methods. One does not need to know the water velocity and the other does not need to know the thickness of the specimen. Because the two through signals are from the receiving transducer, there is no interference of the electric leaking signal. We have applied the two improved methods to characterize doped lead zirconate titanate (PZT-5A) piezoceramics in the frequency range from 25 to 65 MHz. By comparing the measured results with those of the conventional through transmission microscopy method, we conclude that the two improved methods are more convenient and have at least the same accuracy.

II. Characterization Methods

Fig. 1 shows the signal paths in the conventional and the two improved through-transmission ultrasonic spectroscopy methods. Here, S_w refers to the transmitted pulse signal in water (i.e., no specimen in between the two transducers). S_1 and S_2 refer, respectively, to the first and second transmitted pulse signals through the specimen, and d is the thickness of the specimen.

A. Method I

Method I is the conventional method for measuring the frequency dependence of phase velocity and attenuation of

materials. Using this method, two measurements are made. First, the specimen is removed from the wavepath, i.e., the wave travels solely through water medium, and the pulse signal (S_w) is recorded. Then, the specimen is inserted in the wavepath, and the signal (S_1) is recorded. If we assume that the dispersion of water is negligible, the phase velocity [$v(f)$] and attenuation [$\alpha(f)$] of the specimen are obtained by comparing the spectra of Fourier transform of the signals S_w and S_1 [5], [8]:

$$v(f) = \frac{v_w}{1 + \frac{[\varphi_w(f) - \varphi_1(f)]v_w}{2\pi f d}} \tag{1}$$

$$\alpha(f) = \alpha_w + \ln\left[\frac{T(f)A_w(f)}{A_1(f)}\right]/d \tag{2}$$

where v_w and α_w are the sound velocity and attenuation of water, respectively; f is the frequency; φ_w, A_w and φ_1, A_1 are the phase and amplitude spectra of S_w and S_1, respectively; and $T(f)$ is the total transmission coefficient defined as

$$T(f) = \frac{4\rho_w v_w \rho v(f)}{\left[\rho_w v_w + \rho v(f)\right]^2} \tag{3}$$

where ρ_w and ρ are the densities of water and specimen, respectively. In this method, v_w, α_w, and d must be known before the calculations can proceed.

B. Method II

In this improved method, only one measurement is necessary. Two transmitted signals, S_1 and S_2, with specimen in place are recorded. The signal without specimen, i.e., S_w, is not needed. Let φ_2, A_2 be the phase and amplitude spectra of the Fourier transform of S_2; then, the phase velocity and attenuation of the specimen can be obtained in a similar fashion described in [5] and [8]:

$$v(f) = \frac{4\pi d f}{\varphi_1(1) - \varphi_2(f)} \tag{4}$$

$$\alpha(f) = \frac{\ln\left[R(f)A_1(f)/A_2(f)\right]}{2d}. \tag{5}$$

Here, $R(f)$ is the total reflection coefficient, which is defined as

$$R(f) = \frac{\left[\rho_w v_w - \rho v(f)\right]^2}{\left[\rho_w v_w + \rho v(f)\right]^2}. \tag{6}$$

Compared with Method I, here only the specimen thickness (d) is required for the calculation of phase velocity.

If the value of the phase velocity [$v(f_0)$], at a reference frequency f_0 can be predetermined, the need for measuring d can be also eliminated, so that $v(f)$ will be totally determined by the phase spectra $\varphi_1(f)$ and $\varphi_2(f)$:

$$v(f) = v(f_0)\frac{\Delta\varphi_{21}(f_0)f}{\Delta\varphi_{21}(f)f_0} \tag{7}$$

where $\Delta\varphi_{21}(f)$ is defined as

$$\Delta\varphi_{21}(f) = \varphi_2(f) - \varphi_1(f). \tag{8}$$

If the attenuation at a reference frequency is known, we can obtain $\alpha(f)$ without measuring d:

$$\alpha(f) = \alpha(f_0)\left\{1 + \frac{\ln\left[\frac{R(f)A_1(f)A_2(f_0)}{R(f_0)A_1(f_0)A_2(f)}\right]}{\ln\left[R(f_0)\frac{A_1(f_0)}{A_2(f_0)}\right]}\right\}. \tag{9}$$

C. Method III

In this improved method, all three signals, S_w, S_1, and S_2, are recorded. Two measurements are made. The benefit of this method compared with that of [9] is that only transmitted ultrasonic pulses are received by the receiving transducer; there is no influence of the leaking electric signal. One does not need to know the thickness of the specimen in this method.

Combining (1) and (4), we can cancel the thickness d and obtain the phase velocity as

$$v(f) = v_w\left[1 + 2\frac{\varphi_w(f) - \varphi_1(f)}{\varphi_2(f) - \varphi_1(f)}\right]. \tag{10}$$

Substituting (10) into (4), the following expression for the thickness of the specimen can be derived

$$d = \frac{v_w}{4\pi f}\left\{\varphi_2(f) - \varphi_1(f) + 2\left[\varphi_w(f) - \varphi_1(f)\right]\right\}. \tag{11}$$

Substituting (11) into (5), one can get the formula for the attenuation:

$$\alpha(f) = \frac{2\pi f \ln\left[R(f)A_1(f)/A_2(f)\right]}{v_w\left\{\varphi_2(f) - \varphi_1(f) + 2\left[\varphi_w(f) - \varphi_1(f)\right]\right\}}. \tag{12}$$

Similarly, if $v(f_0)$ can be predetermined, the need for measuring v_w will be eliminated, then $v(f)$ is totally determined by the phase spectra $\varphi_w(f)$, $\varphi_1(f)$, and $\varphi_2(f)$:

$$v(f) = v(f_0)\frac{1 + 2\Delta\varphi_{w1}(f)/\Delta\varphi_{21}(f)}{1 + 2\Delta\varphi_{w1}(f_0)/\Delta\varphi_{21}(f_0)} \tag{13}$$

where $\Delta\varphi_{w1}(f)$ is defined as

$$\Delta\varphi_{w1}(f) = \varphi_w(f) - \varphi_1(f) \tag{14}$$

and $\alpha(f)$ is given by [(15), see next page], assuming $\alpha(f_0)$ is known.

The data processing in all of these methods is performed in the frequency domain, which is different from the tradition time-of-flight methods [10], [11] where the data processing is performed in time domain.

$$\alpha(f) = \alpha(f_0)\frac{\ln\left[R(f)A_1(f)/A_2(f)\right]\cdot f\cdot\left[\Delta\varphi_{21}(f)+2\Delta\varphi_{w1}(f)\right]}{\ln\left[R(f_0)A_1(f_0)/A_2(f_0)\right]\cdot f_0\cdot\left[\Delta\varphi_{21}(f_0)+2\Delta\varphi_{w1}(f_0)\right]} \tag{15}$$

III. Experimental Results and Discussion

The basic setup for immersion ultrasonic experiments has been described in detail in [7], [8]. Two immersion-type broadband transducers (Panamatrix V358) with center frequency of 50 MHz and bandwidth of 80% are used. The transmitting transducer (T in Fig. 1) is driven by a pulser (Panametrics 5052PR) that produces short-duration (5 ns) pulses with a repetition rate of 1 kHz. The signal from the receiving transducer (R in Fig. 1) is sampled using a digital oscilloscope (Tektronix TDS 460A), which has an adjustable digital delay for triggering the sampling window. The sampling rate is set to 10 Gs/s to avoid aliasing. Each sampling window contains 2500 data points. To reduce the ubiquitous random errors, each measurement is averaged 64 times. The data are transferred to a computer via a general purpose interface bus (GPIB). The fast Fourier transform (FFT) is then performed on the 8192 data points by adding zeros at the end of the signal, which gives a frequency resolution of about 1 MHz. The water temperature is maintained at $21.3 \pm 0.1°C$, so that the water velocity is taken as 1486 m/s from [12].

A plate with surface dimension of 2.5 cm x 2.5 cm PZT-5A piezoceramic (ValpeyFisher, Hopkinton, MA) was used as the test sample. The thickness of the PZT-5A specimen is 1.088 ± 0.003 mm measured at five different locations across the sample surface using a digital indicator (Starrett 2720).

The three received pulse signals, S_w, S_1 and S_2, are shown in Fig. 2(a, b, and c). Their phase and amplitude spectra are shown in Fig. 3(a, b, and c). The phase velocity and attenuation in the frequency range from 25 to 65 MHz are calculated for PZT-5A using the conventional method (squares) and the two improved methods (circles and upper triangles) after the trigger delay time is taken into account. The results are shown in Fig. 4 and 5. It is found that the three methods produce a discrepancy of $< 1\%$. For example, the discrepancy in phase velocity is only 0.3% between Methods II and I and 0.7% between Methods III and I. The value of phase velocity is slightly higher than the value in low frequency [13] as expected.

The uncertainty of water velocity used in the calculation is the main source for the discrepancy between Methods II and I because the sound velocity in water varies with the degree of purity and the reported book value did not specify the water purity. If we substitute the phase velocity obtained from Method II into $v(f)$ in (1), the sound velocity in water can be estimated to be 1488 m/s, which is the same as our direct measurement, but a little higher than the cited book value from [12]. Two different values of the sample thickness used in the calculations are the main sources for the discrepancy between Methods III and I. A better agreement could be expected if only one thickness value is used. In fact, if we increase the mechanically measured thickness to the calculated value, the curves in Methods II and I almost overlap exactly with each other.

This observation suggests that the newly introduced Method II is preferred based on its simplicity because the data reduction can be done from a single experiment if the second transmitted pulse signal S_2 is strong enough. On the other hand, based on the theory of error propagation in the measurement uncertainties, Mobley proved that, in ultrasonic spectroscopy method, the thickness uncertainty dominates when the specimen and water speeds are significantly different [6]. Therefore, Method III will be the most accurate one because it can eliminate the thickness uncertainty. This method could also be used in the nondestructive evaluation of structural components where the thickness of the object is difficult to measure directly or varies spatially. Such a feature is also useful in the ultrasonic evaluation of biological tissues, where flat and uniform specimens are difficult to obtain. On the other hand, Methods II and III do not work well for very high attenuation materials because the acoustic path length of signal S_2 is three times the sample thickness; therefore, the signal will be too small to be used because of high attenuation.

A comparison of the mechanically measured thickness with that determined ultrasonically using (11) is shown in Fig. 6. The agreement is within the measurement error as indicated by the error bar in Fig. 6. The flatness of the curve (0.02% standard derivation) provides a strong support to the reliability of the proposed Method III. In theory, the frequency dependence of all of the components on the right side of (11) should be completely canceled out, making d a frequency-independent quantity.

IV. Conclusion

Two improved through-transmission ultrasonic spectroscopy methods are investigated for the determination of the dispersion of phase velocity and attenuation of solid materials. For each method, the equations for the calculation of phase velocity and attenuation are derived. The new methods were applied to measure the ultrasonic dispersion of velocity and attenuation of PZT-5A in the frequency range from 25 to 65 MHz, and the results agree well with that obtained from the conventional method (Method I). The two new methods require one less predetermined parameter and are, therefore, more convenient to use and eliminate one more error source. However, they may not be used for extremely attenuate materials for which the S_2 is too small to be detected.

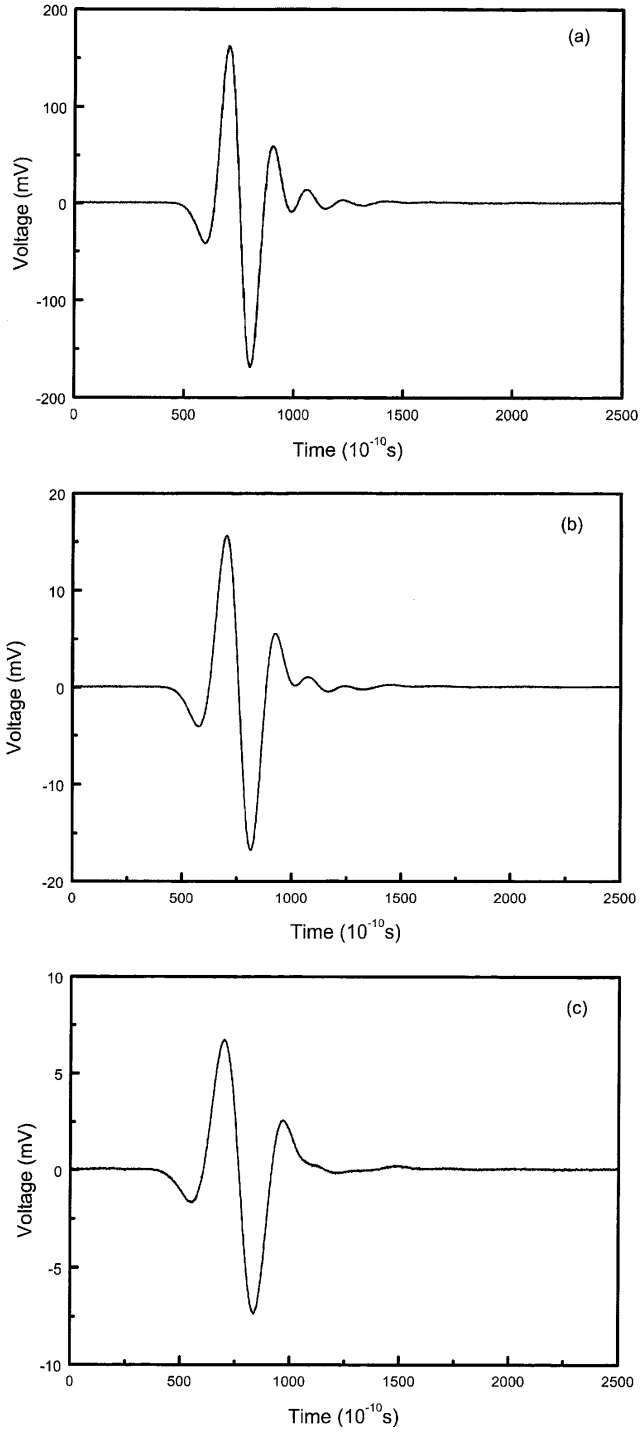

Fig. 2. Three received pulse signals: a) S_w, the transmitted pulse signal in water; b) S_1, the first transmitted pulse signal through the specimen; and c) S_2, the second transmitted pulse signal through the specimen.

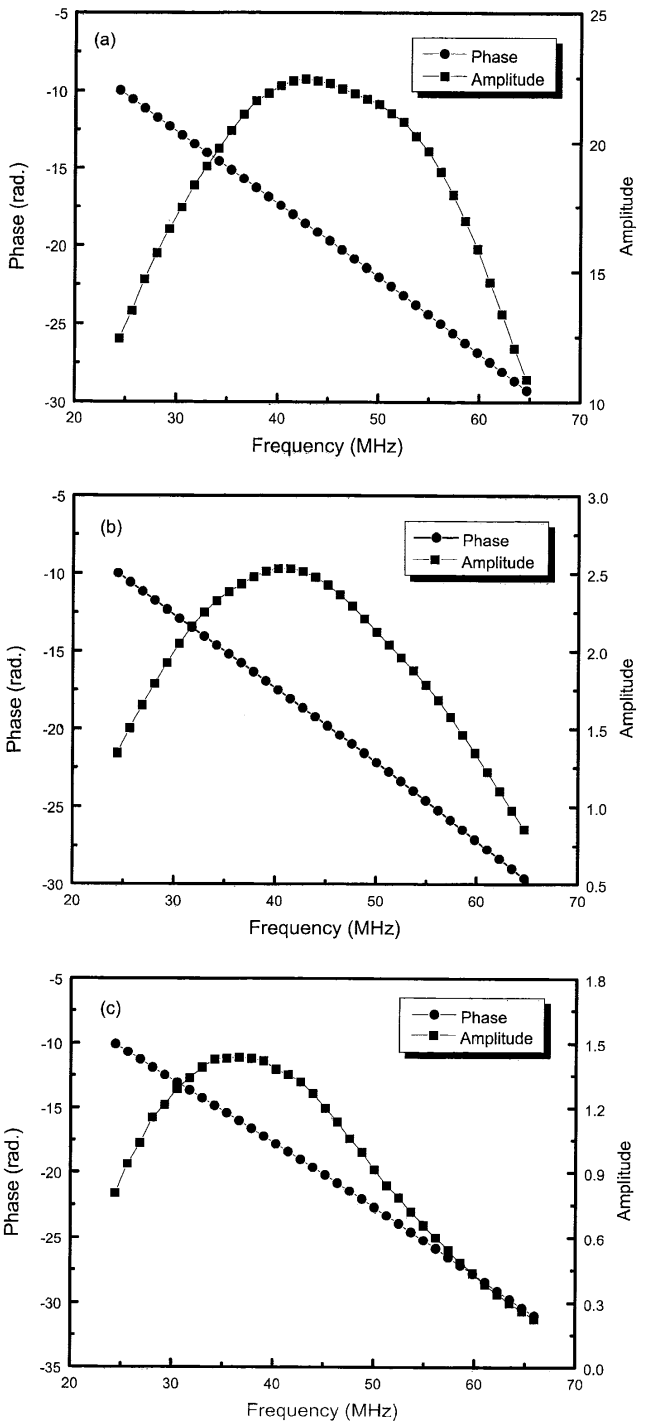

Fig. 3. The phase and amplitude spectra of the three pulse signals shown in Fig. 2: a) the spectra of S_w, b) the spectra of S_1, and c) the spectra of S_2.

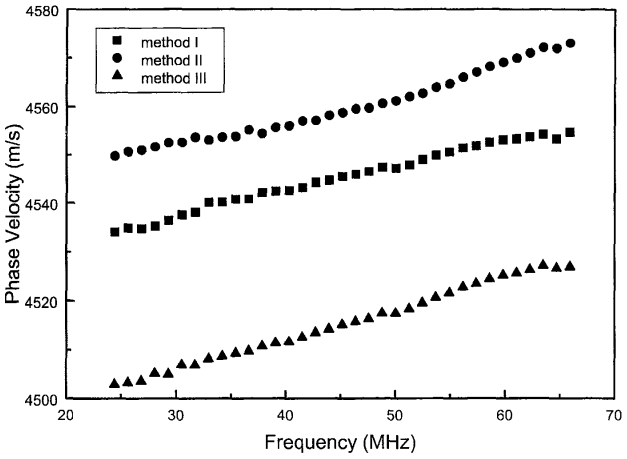

Fig. 4. Frequency dependence of phase velocity in PZT-5A determined using all three methods [Method I(■), Method II(●), and Method III(solid triangle)].

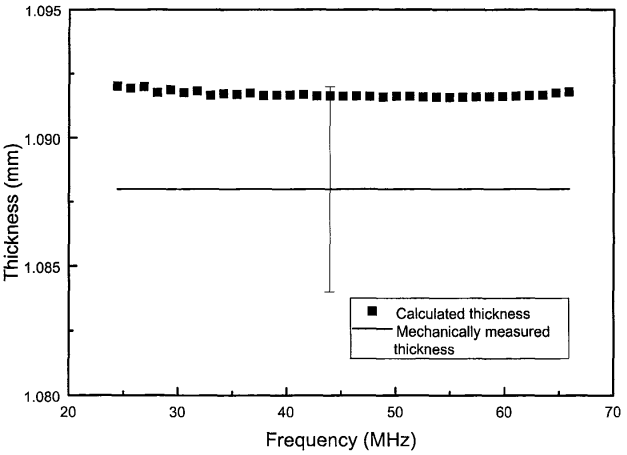

Fig. 6. A comparison between the mechanically measured thickness with that determined ultrasonically using Method III.

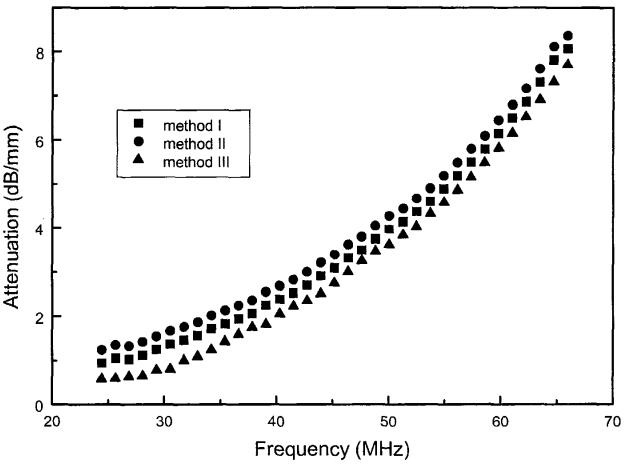

Fig. 5. Frequency dependence of attenuation in PZT-5A determined using three methods [Method I(■), Method II(●), and Method III(solid triangle)].

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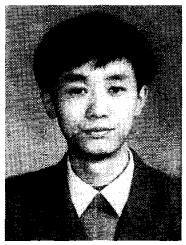

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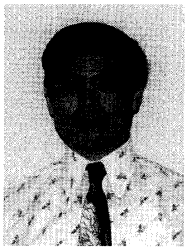

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