AN ULTRASONIC METHOD FOR SIMULTANEOUS DETERMINATION OF ELASTIC MODULI, DENSITY, ATTENUATION AND THICKNESS OF A POLYMER COATING ON A FOIL

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INTRODUCTION

Ultrasonic spectroscopy has for a long time been thought promising for characterization of thin layers immersed in water or embedded between two known materials (similar or dissimilar) [1]. Significant effort has been put forth by many authors [2]–[14]. An ultrasonic spectroscopy method for determination of the complete set of acoustical and geometrical properties of an isotropic layer embedded between two known materials (similar or dissimilar) has been developed recently in [15]. The uniqueness of the method is its ability to determine all the layer properties – thickness, density, elastic moduli and attenuation (longitudinal and shear) – at two incident angles: one normal and one oblique.

Ultrasonic signals reflected from the front and back surfaces of a thin layer usually overlap in the time domain and interfere. In practice, the layer can be attached to one or two solid substrates (as, for example, in ceramic coated metal plates or in adhesive joints). In our previous work [15], thick substrates were considered and the signal reflected from the bonding layer separated in the time domain from the signals reflected from the front and back surfaces of the substrates. In some practical systems the substrates are thin, as in examples considered in this paper for a polymer coated steel or aluminum foils with total thickness of the system of 100–200 μm. In this case the interface signal cannot be separated in the time domain from the front and back surface reflections even if transducers with 30-50 MHz bandwidth are used. For these systems the reflection (transmission) signal is formed by interference of multiple wave reflections inside the laminate layers.

In this paper we extend the ultrasonic method developed in [15] to the coated foils. The method allows one to determine properties of the coating layer – density, thickness and longitudinal and shear moduli and attenuations – using ultrasonic measurements at only two angles: one normal and one oblique. The input data for inversion are two reflection spectra of the interference signals formed by multiple reflections of the ultrasonic wave inside the two-layer system. We assume the bond between layers perfect, the foil properties known and the coating layer isotropic.

In the first section characteristic features of ultrasonic wave interaction with a coated foil are discussed. A method for determination of the polymer coating properties is introduced in the second section. The experimental results are described in the third section. An

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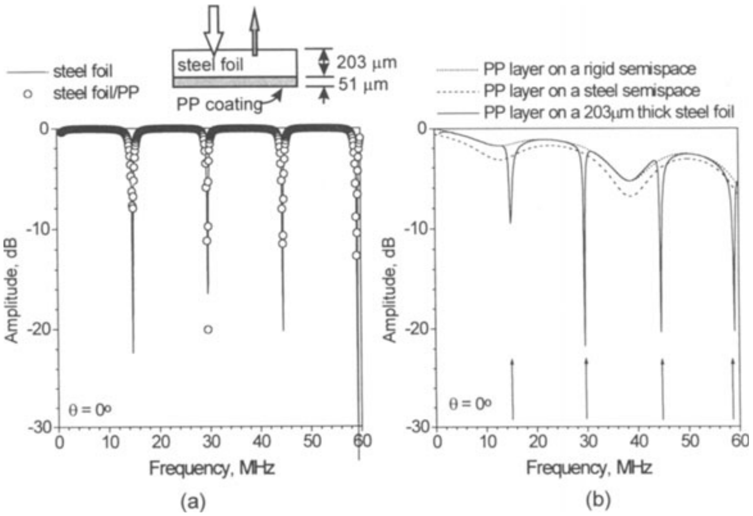


Figure 1. (a) Normal incidence reflection spectra for the PP coated steel foil, (reflection from steel side) and for a steel foil; (b) Normal incidence reflection spectra for the PP coating on a rigid semispace (solid line), on a steel semispace (dashed line) and on a steel foil (short dashed line).

example of experimental results presented in this paper is for a 51 μm thick polypropylene (PP) film bonded to a 203 μm steel foil. This type of coating is used in food containers. The parameters of the PP coating must be within specified limits to ensure food encapsulation.

CHARACTERISTIC FEATURES OF ULTRASONIC WAVE INTERACTION WITH A COATED FOIL

An important feature of polymer coated steel metal foil (in our example) is that the impedance of metal is more than ten fold that of a polymer. This impedance difference results in interesting features of the spectra of the signals reflected from the steel and from the polymer side. Figure 1(a) shows calculated reflection spectra at normal incidence for steel foil and for the same foil coated by PP on the back side (reflection is from the steel side). One can see that the spectra are practically indistinguishable. The steel foil vibration is almost undisturbed by a low impedance coating on the side opposite to the excitation. In this case, due to much higher impedance of steel compared to this of the polymer coating only a small fraction of energy penetrates the PP layer and since the impedances of PP and water are similar most of the energy penetrated PP layer is transmitted into water. This indicates, as will be discussed later, that the signal reflected from steel side of the coated steel foil carries little information on the coating properties.

The calculated reflection spectrum for the ultrasonic wave incident from the coating side is shown in Figure 1(b). The results are very close for a PP coating on a steel foil (solid line) and on an infinitely rigid semispace (short dash line). Due to small impedance difference between water and polypropylene the minima are broad and shallow. Since the PP layer is embedded between media with impedances lower and higher than that of polypropylene the broad minima correspond to resonance condition: $h_{PP} = \lambda_{PP}/4 + n \cdot \lambda_{PP}/2, n = 0, 1, 2, \ldots.$ Deep and narrow resonance minima observed for a coating/foil system correspond to steel/foil laminate resonance (marked by arrows in Fig. 1b). Since the steel foil impedance is much greater than that of polypropylene and water the minima approximately correspond to resonance condition: $h_{steel} = \lambda_{steel}/2, n = 0, 1, 2, \ldots.$

For comparison Figure 1(b) also shows the spectrum for a PP layer on a steel semispace (long dashed line). While the minima positions are the same as for the PP layer on the

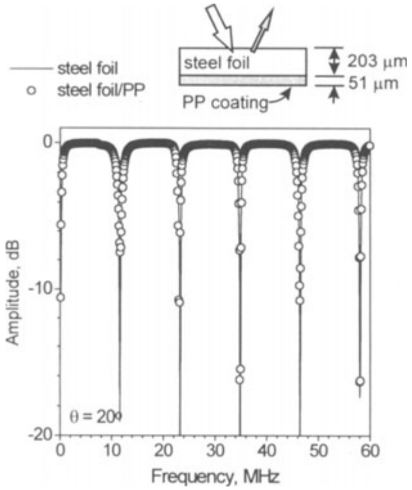


Figure 2. Reflection spectra at 20° for the PP coated steel foil (reflection from steel side) and for a steel foil.

rigid semispace the reflection amplitude is lower due to partial energy loss with transmission through the layer. The reflection amplitude for a PP coating on a steel foil is close to that of a PP coating on a rigid (not steel) semispace since most of the energy transmitted into steel foil is reflected back at the steel/water interface, which takes part of the energy away from the coating..

Analogous phenomena are observed at oblique incidence ($\theta_0 = 20°$). Figure 2 shows oblique incidence reflection spectra from a steel foil and from a foil coated by PP on the back side. One can see that the difference between the spectra is very small which suggests low sensitivity of the signal reflected from the PP coated foil (from steel side) to the polypropylene properties.

When the ultrasonic wave is incident from on the coated foil from the coating side, PP layer vibration is similar to this of the PP layer on the rigid semispace as demonstrated by the reflection spectra shown in Figure 3. For a PP on a thin foil the minima corresponding to the foil resonance appear (marked by arrows: 12, 24, 36 MHz, etc.). It is important to note that the spectrum for oblique incidence from the coating side (on the contrary to incidence from steel side and to spectrum at normal incidence) has deep and narrow characteristic minima related to the coating layer resonances (6, 25, 32 and 48 MHz) (short dash lines in Figures 3 and 1(b)). Examples of experimental measurement from PP and steel side in a low frequency range are shown in Figures 4a,b. One can see that while the steel plate resonance is observed from both sides at about 12 MHz the resonance due to PP layer (6 MHz) is observed only from the coating side. As will be shown below, the frequency ranges near PP layer resonance minima are sensitive to the coating properties. The minimum near 6 MHz has great practical importance since it allows determining polypropylene coating properties using a conventional low frequency transducer.

DETERMINATION OF LAYER PROPERTIES FROM ULTRASONIC MEASUREMENTS

Definition of the Unique Set of Material Parameters

The observations of the previous section show that at certain conditions the signals reflected from the coated foil are sensitive to the coating properties. To determine these properties we extend methodology described by us in [15] for characterization of a layer embedded between thick substrates.

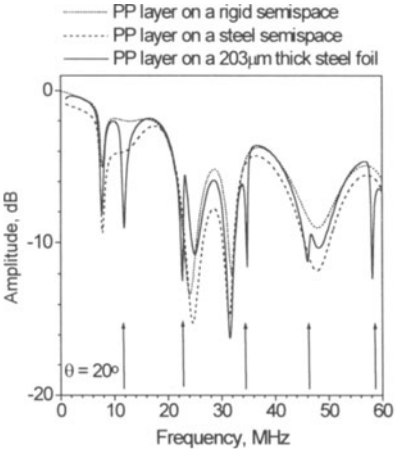
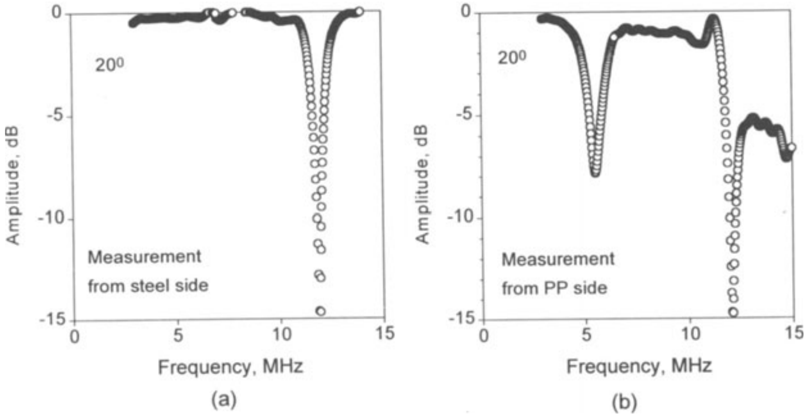


Figure 3. Reflection spectra at 20° for the PP coating on a rigid semispace (solid line), on a steel semispace (dashed line) and on a steel foil (short-dashed line).



(a)

(b)

Figure 4. Spectra of reflection from the polypropylene coated steel foil at 20° from a) steel side, b) polypropylene side.

It has been shown in [15] that the number of parameters describing the normal incidence reflection (transmission) coefficient from the layer can be reduced from four (elastic modulus, density, thickness and attenuation) to three nondimensional parameters: impedance ratio Z_N, nondimensional thickness $\overline{h}$ and attenuation α_ℓ, given by

$$Z_N = \frac{Z_2}{Z_1}, \ \overline{h}_\ell = \frac{h}{V_\ell}\omega_0, \ \alpha_\ell = k''_\ell / k'_\ell, \tag{1}$$

Here $V_\ell = [(\lambda + 2\mu)/\rho]^{1/2}$ is the longitudinal velocity in the adhesive layer, $\omega_0 = 1\,\mathrm{MHz}$.

At oblique incidence both longitudinal and shear waves are excited inside the layer. Reflection and transmission coefficients depend on six layer parameters: elastic moduli, thickness, density, and longitudinal and shear wave attenuations:

$$\lambda + 2\mu, \ \mu, \ \rho, \ h, \ \alpha_\ell, \ \alpha_t. \tag{2}$$

A full set of nondimensional parameters defining oblique incidence reflection and transmission includes Z_N, $\overline{h}_\ell$ and α_ℓ and three more nondimensional parameters: two nondimensional thicknesses $\overline{h}_{\theta\ell}$ and $\overline{h}_{\theta t}$ at oblique incidence and shear wave attenuation α_t

$$\overline{h}_{\theta\ell} = \frac{h\cos\theta_\ell}{V_\ell}\omega_0, \ \overline{h}_{\theta t} = \frac{h\cos\theta_t}{V_t}\omega_0, \ \alpha_t = k''_t / k'_t, \tag{3}$$

where $V_t = [\mu/\rho]^{1/2}$ is the shear wave velocity in the adhesive layer, θ_ℓ, θ_t are longitudinal and shear wave propagation angles inside the layer, and $\omega_0 = 1\,\mathrm{MHz}$ is introduced to satisfy nondimensionality. All six nondimensional parameters can be found by the inversion algorithm described below from two measurements: one at normal and the other at oblique incidence. Four nondimensional parameters from the sets (1, 3) $(Z_N, \overline{h}_\ell, \overline{h}_{\theta\ell}, \overline{h}_{\theta t})$ fully define the dimensional parameters from the set (2):

$$\lambda + 2\mu = \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}{\overline{h}_\ell}, \tag{4}$$

$$\mu = \frac{Z_N Z_1}{\xi_0} \frac{\overline{h}_\ell \sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2 + \overline{h}_{\theta t}^2}, \tag{5}$$

$$\rho = Z_N Z_1 \xi_0 \frac{\overline{h}_\ell}{\sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}, \tag{6}$$

$$h = \frac{\sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}{\xi_0 \omega_0}, \tag{7}$$

where $\xi_0 = \sin\theta_0 / V_0$, and θ_0, V_0 are propagation angle and wave velocity in water. Attenuations α_ℓ and α_t are part of both nondimensional (1, 3) and dimensional (2) sets of parameters. Instead of the elastic moduli, the longitudinal and shear wave velocities, V_ℓ and V_t, can be used. They are related to the nondimensional parameters by

$$V_\ell = \frac{\sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}{\xi_0 \overline{h}_\ell}, \ V_t = \frac{\sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2}}{\xi_0 \sqrt{\overline{h}_\ell^2 - \overline{h}_{\theta\ell}^2 + \overline{h}_{\theta t}^2}}. \tag{8}$$

Inversion Algorithm

As discussed in the previous section, the reflection spectrum at normal incidence is fully defined by three nondimensional parameters: Z_N, $\overline{h}_\ell$ and attenuation α_ℓ, which can be determined from experimental data by inversion. Z_N and $\overline{h}_\ell$ are functions of three dimensional parameters: $\lambda + 2\mu, h$ and ρ. Two of them can be determined only if the third is known: this is the main limitation of using only normal incidence measurements for layer characterization. To determine all layer properties (2) oblique incidence measurements are also required.

It was proposed in [15] to use measurements of both normal and oblique incidence reflection (transmission) spectra for determining six layer properties (2). For the nondimensional set of parameters (1, 3) the problem is factorized (decomposed) to use of the two-step algorithm for determination of the coating properties in a three-dimensional space of parameters. First, we determine three nondimensional parameters: Z_N, $\bar{h}_\ell$ and α_ℓ, from reflection (transmission) spectra at normal incidence. Next, considering Z_N, $\bar{h}_\ell$ and α_ℓ to be known, three more nondimensional parameters of the coating $\bar{h}_{\theta\ell}$, $\bar{h}_{\theta t}$, α_t are determined from oblique incidence data (reflection or transmission) . The corresponding dimensional parameters (2) are calculated using equations (4)–(7). The foil properties are considered to be known.

For inversion we employ the least squares method for the minimization of the sum of squared deviations between experimental and calculated reflection (transmission) coefficients considering nondimensional parameters as variables in a multidimensional space:

$$\min_{X_i \in \Re^n} \frac{1}{2} \sum_{i=1}^{m} (|R_i^e| - |R_i^c|)^2 \tag{9}$$

Here X_i are nondimensional parameters, $n = 3$ is the number of parameters to be found, m is the number of data points at different frequencies, and R^e and R^c are the experimental and calculated reflection (transmission) coefficients, respectively. It was shown [15] that the proposed method (in application to the polystyrene layer between aluminum substrates) is robust against experimental noise in the time-domain signal.

Application of the Inversion Algorithm for Characterization of the Coated Foils

To characterize a foil coating we use the reflected interference signal formed by reverberations of the ultrasonic wave in both coating and substrate layer as an input for the inversion problem. The effect of the coating layer on the reflected signal is smaller than in the case of interphase layer between semispaces and depends on foil properties. However, certain frequency ranges of the measured spectra (certain resonance modes of the laminate) are fairy sensitive to the layer properties. Thus, to determine the layer parameters with maximum precision the 'sensitive' frequency ranges was found.

EXPERIMENT

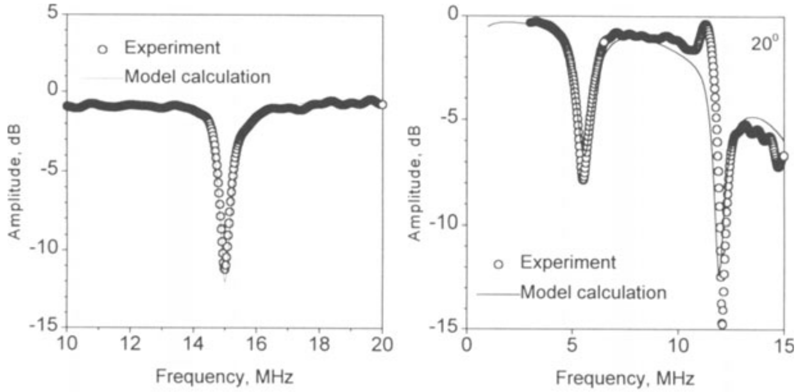
Angle–Beam Reflection Technique

The angle–beam reflection measurements were performed using a specially developed ultrasonic goniometer [16], which can focus an obliquely incident ultrasonic wave on the coated foil by shifting the position of the foil along the cylinder radius. The goniometer is unique in its ability to measure reflected interfacial signals at various incident angles with only one transducer. We used the angle of wave incidence on the coated foil greater than the first critical angle of 14.8°.

The experiments have been done using an ultrasonic experimental system [5] controlled by a computer. The reflected ultrasonic signals were amplified, digitized, averaged by a HP 54504A 400 MHz digital oscilloscope, and collected by an IBM AT through an IEEE–488 interface. The data is then processed in the frequency domain using an FFT program and deconvolved with a reference signal taken from the aluminum–air interface (for the deconvolution a special sample with air gap was used). The rotation of the transducer, i.e. change of incident angle, and position of a sample using a translation table are controlled by a PC AT through an RS–232 interface using a CTC–283 DC Motor Controller, manufactured by Micro Kinetics Co. Resolution for the rotation table is 0.01° and for the translation table is 0.01 mm. The whole apparatus was immersed in water with temperature stabilized at 29.8± 0.02°C using a Fisher Isotemp Circulator (Model 730).

Experimental Determination of the Full Set of the PP Coating Properties

The properties of the polypropylene coating on to the steel foil were determined using a



(a) (b)

Figure 5. Spectra of reflection from the polypropylene coated steel foil at a) 0°, b) 20°.

Table 1. Comparison of properties of the polypropylene coating determined ultrasonically and by direct measurements.

	$\lambda + 2\mu$, GPa	μ, GPa	ρ, g/cm^3	h, mm	α_ℓ	α_t
Ultrasonic measurement	6.27	1.28	1.02	0.056	0.020	0.064
Reference data	6.30*		0.935**	0.051**		
* — [20], ** — direct measurement						

combination of normal/oblique incidence measurements. The measurements were performed at 0°, 20°, 21° and 22° by the angle-beam reflection technique using ultrasonic transducers with central frequencies at 5 and 10 MHz and corresponding bandwidths (at -10 dB level) of 5 and 10 MHz respectively. Data at only two angles were used simultaneously for reconstruction. Figure 5 shows the spectra for 0° and 20°. Three nondimensional parameters, Z_n, $\overline{h}_\ell$ and α_ℓ, were determined from the reflection spectra at normal incidence. Three more nondimensional parameters, $\overline{h}_{\theta\ell}$, $\overline{h}_{\theta t}$, and α_t, were determined for reflection at each of the oblique incidence angles with Z_N, $\overline{h}_\ell$ and α_ℓ taken as known. The corresponding dimensional parameters (2) were calculated using equations (4)–(7). The solid lines in Figure 5 represent spectra calculated from the parameters determined. Similar results were obtained for 21° and 22° incidence angles; these results were averaged.

Table 1 compares the film properties determined ultrasonically to the moduli values from [20] and to the directly measured thickness and density (i.e. thickness measured by micrometer and density by a water displacement method).

The difference between ultrasonic and direct measurement of the polypropylene film thickness and density is less than 10%. The comparison is reasonable taking into account hat direct measurement have several percent error.

SUMMARY AND CONCLUSIONS

This paper describes an ultrasonic method for determining the properties of an isotropic polymer coating on a thin foil (thickness less than the ultrasonic wavelength). The method allows simultaneous determination of all coating properties — thickness, density, longitudinal and shear elastic moduli and attenuations — from two measured reflection spectra: one at normal and one at oblique incidence. The substrate properties (metal foil) are assumed to be known.

By introducing nondimensional parameters the number of parameters describing the normal incidence reflection (transmission) coefficient is reduced from four to three. The minimal set of six nondimensional parameters formed from the layer properties fully describes the reflection (transmission) at oblique incidence. By introducing nondimensional parameters the problem is simplified by using instead of inversion in a six-dimensional space (for the six parameters) two consecutive inversions in three-dimensional spaces: for three parameters from the normal incidence data and for the remaining three parameters from the oblique incidence data. This two-step inversion algorithm is more robust against error in experimental data. The thickness, density and elastic moduli of the layer are calculated from the nondimensional parameters determined using simple analytical expressions.

The sensitivity of the proposed method to the individual properties and the stability of the method against experimental noise are studied for a 56 μm polypropylene coating on a thin steal foil. The 'sensitive' frequency ranges are identified and the algorithm is optimized accordingly.

The properties of the polypropylene coating on the steel foil were determined experimentally which are in reasonable agreement with those determined by direct measurements.

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