

ULTRASONIC MEASUREMENT OF THICKNESS, DENSITY AND ELASTIC MODULI OF A LAYER EMBEDDED INTO A SOLID

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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). Significant effort has been put forth by many authors.

Ultrasonic signals reflected from the front and back surfaces of a thin layer usually overlap in the time domain and interfere. Flynn [1] and Chang *et al* [2] determined ultrasonic velocity and attenuation from the ultrasonic reflected signal and correlated them with the joint cohesive strength. The influence of frequency dependent attenuation on amplitude and phase spectra of the signal reflected from the joint bondline was studied in [3]. Through-thickness resonance measurements were used in [4, 5] to calculate both the thickness and the modulus of an adhesive layer. A low frequency through-transmission ultrasonic technique was proposed in [6] to determine the thickness of a thin paper or metal layer. Inversion of leaky Lamb wave dispersion curves was used in [7] to determine longitudinal and shear wave velocities and thickness of an adhesive layer inside joint. An ultrasonic technique for evaluation of thin layers and adhesive joints was proposed in [8]–[11]. The authors were able to reconstruct from the normal incidence reflection or transmission one of the parameters (velocity, density or thickness) when the others were known. Normalized amplitude spectra were used in [12] to measure attenuation. A review of adhesive joint testing is given in [13].

In this paper we propose an ultrasonic method for simultaneous determination of all properties of the layer placed between two solids. To do this we perform ultrasonic measurements at two angles: normal and oblique incidence. We assume the adhesive/adherend bond is perfect, the adherend properties known and the adhesive isotropic. In the first section the ultrasonic wave interaction with a layer is described by six nondimensional parameters. An inversion algorithm to determine the layer properties is introduced and stability of the method against random noise is studied. The second section describes experiments to validate the method.

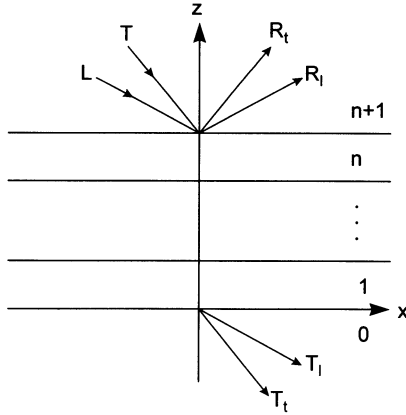


Figure 1. Schematic of ultrasonic wave reflection and transmission through a layered medium.

THEORY

Reflection and Transmission Through the Layer

Consider an ultrasonic wave incident on a multilayered structure as shown in Figure 1. To calculate the ultrasonic wave reflection (transmission) coefficient, a matrix algorithm can be used [14, 15, 16]. The matrix equation for reflection and transmission coefficients can be written in the form

$$\begin{bmatrix} A_\ell \\ R_\ell \\ A_t \\ R_t \end{bmatrix} = [D] \begin{bmatrix} T_\ell \\ 0 \\ T_t \\ 0 \end{bmatrix}, \quad (1)$$

where R_l, R_t are the reflection coefficients of the longitudinal and transverse waves respectively, T_l, T_t are the corresponding transmission coefficients, A_ℓ, A_t are the normalized potential amplitudes of the incident longitudinal and transverse waves: $A_\ell = 1, A_t = 0$ for longitudinal incident wave and $A_\ell = 0, A_t = 1$ for transverse. The matrix D is:

$$[D] = [B^{(n+1)}][a^{(n)}][a^{(n-1)}] \dots [a^{(2)}][a^{(1)}][A^{(0)}]. \quad (2)$$

The matrices $A^{(0)}$ and $B^{(n)}$ relate to the solid semispaces. Isotropic and anisotropic layers are represented by 4×4 matrices $[a^{(i)}]$ ($i = 1, \dots, n$, where n is the number of layers) whose elements are given in [15, 16].

In this paper we consider a layer embedded between two semispaces (i.e. $n = 1$). Our goal is to determine the properties of the layer.

Definition of the Unique Set of the Material Parameters

At oblique incidence both longitudinal and shear waves are excited inside the layer, and thus reflection and transmission depend on six layer properties: elastic moduli $(\lambda + 2\mu)$ and μ , thickness h , density ρ , and longitudinal and shear wave attenuations.

We introduce six nondimensional parameters: normalized impedance $Z_N = Z_2/Z_1$, nondimensional thickness at normal incidence $\bar{h}_\ell = \omega_0 h/V_\ell$, nondimensional thicknesses at oblique incidence $\bar{h}_{\theta\ell} = \omega_0 h \cos \theta_\ell/V_\ell$ and $\bar{h}_{\theta t} = \omega_0 h \cos \theta_t/V_t$, and longitudinal and shear wave attenuations $\alpha_\ell = k_\ell''/k_\ell'$, and $\alpha_t = k_t''/k_t'$, where Z_1, Z_2 are impedances of the semispace material and the layer, $V_\ell = [(\lambda + 2\mu)/\rho]^{1/2}$, $V_t = [\mu/\rho]^{1/2}$ are longitudinal and shear wave velocities in the layer, θ_ℓ, θ_t are corresponding propagation angles, and $\omega_0 = 1$ MHz.

The set of six nondimensional parameters:

$$Z_N, \bar{h}_\ell, \bar{h}_{\theta\ell}, \bar{h}_{\theta t}, \alpha_\ell, \alpha_t, \quad (3)$$

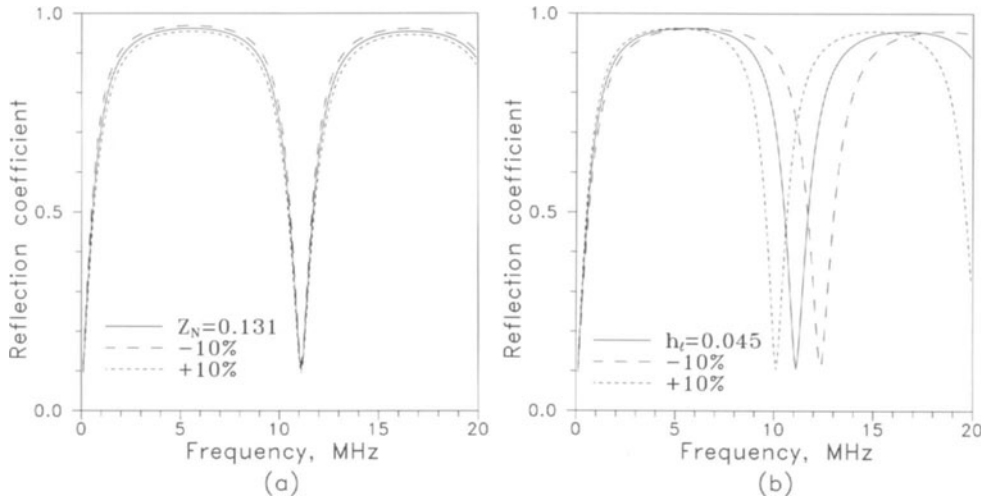


Figure 2. Effect of a) normalized impedance Z_N and b) nondimensional thickness \bar{h}_ℓ on reflection from a polymer layer between aluminum plates. Parameters used for calculation are: $Z_N=0.131$, $\bar{h}_\ell=0.045$, $\alpha_\ell=0.01$.

fully describes reflection from (transmission through) the layer; it is equivalent to:

$$\lambda + 2\mu, \mu, \rho, h, \alpha_\ell, \alpha_t. \quad (4)$$

The dimensional parameters (4) are related to the nondimensional parameters (3) by

$$\lambda + 2\mu = \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}{\bar{h}_\ell}, \quad (5)$$

$$\mu = \frac{Z_N Z_1}{\xi_0} \frac{\bar{h}_\ell \sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2 + \bar{h}_{\theta t}^2}, \quad (6)$$

$$\rho = Z_N Z_1 \xi_0 \frac{\bar{h}_\ell}{\sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}, \quad (7)$$

$$h = \frac{\sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}{\xi_0 \omega_0}, \quad (8)$$

where $\xi_0 = \sin \theta_0 / V_0$, and θ_0, V_0 are propagation angle and wave velocity in water. Longitudinal and shear wave velocities, V_ℓ and V_t , are related to the nondimensional parameters by

$$V_\ell = \frac{\sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}{\xi_0 \bar{h}_\ell}, \quad V_t = \frac{\sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2}}{\xi_0 \sqrt{\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2 + \bar{h}_{\theta t}^2}}. \quad (9)$$

Example for Normal Incidence

At normal incidence reflection and transmission coefficients depend only on Z_N, \bar{h}_ℓ and α_ℓ . Figure shows example calculations of the effect of the nondimensional parameters on reflection spectrum. Calculations are done using parameters typical for a polymer layer between aluminum semispaces. The minima observed in the reflection spectra are due to destructive interference of the wave reflected from the front surface of the layer and waves multiply reflected inside the layer. One can see that for fixed α_ℓ and \bar{h}_ℓ increase of the impedance mismatch Z_N results in reflection amplitude decrease (Fig. a). At fixed α_ℓ and Z_N nondimensional thickness \bar{h}_ℓ increase results in minima shift towards lower frequency (Fig. b) while not changing the maxima and minima amplitudes.

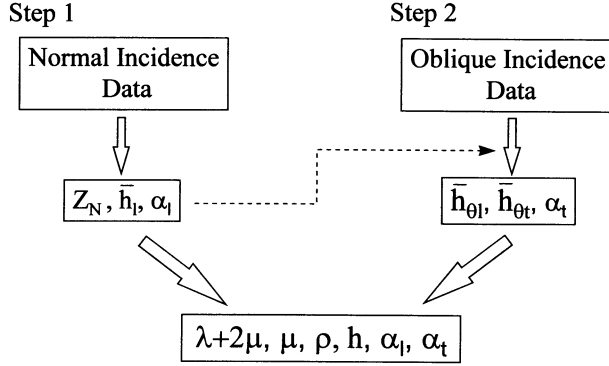


Figure 3. Schematic of the algorithm for determination of adhesive layer properties from normal and oblique incidence reflection (transmission) spectra.

ALGORITHM FOR DETERMINATION OF THE LAYER PROPERTIES

The shown example demonstrates that the reflection spectrum is fully defined by two nondimensional parameters: Z_N and \bar{h}_ℓ (and attenuation) which can be determined from experimental data by inversion. Z_N and \bar{h}_ℓ 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 the layer characterization. To determine all layer properties (4) oblique incidence measurements are also required.

We propose an algorithm for determination of layer properties (Eq. (4)) without prior knowledge of any of them using measurements of both normal and oblique incidence reflection spectra. 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 (3) as variables in a multidimensional space:

$$\min_{X_i \in \mathbb{R}^n} \frac{1}{2} \sum_{i=1}^m (|R_i^e| - |R_i^c|)^2 \quad (10)$$

Here X_i are nondimensional parameters (3), n 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.

Oblique incidence reflection (transmission) coefficients depend on six adhesive layer parameters ((3) or (4)). In practice one finds that all six parameters cannot be reconstructed from a single oblique incidence measurement. We propose a two-step algorithm for determination of the adhesive layer properties (Fig. 3). First, we reconstruct three nondimensional parameters: Z_N , \bar{h}_ℓ and α_ℓ , from reflection (transmission) spectra at normal incidence. Second, three more nondimensional parameters $\bar{h}_{\theta\ell}$, $\bar{h}_{\theta t}$, α_t are determined from oblique incidence data (reflection or transmission) with Z_N , \bar{h}_ℓ and α_ℓ taken as known. The corresponding dimensional parameters (4) are calculated using equations (5)–(8).

Stability of the Inversion Algorithm to the Scatter in the Experimental Data

We can characterize the accuracy of the determined parameters by studying the stability of the inversion procedure against random noise in the input time-domain signal. In our numerical simulation a set of polymer film properties ('original set') is used to generate synthetic reflection spectra at normal and oblique incidence. The spectra are overlapped with a typical transducer spectrum and a backward FFT procedure is used to calculate the corresponding synthetic time-domain signal. Next, random noise is introduced into these signals (Fig. 4a) to simulate possible experimental noise. A forward FFT procedure is applied to the obtained 'noisy' time-domain signal and the resulting frequency-domain signals are deconvolved with the transducer spectrum to obtain a synthetic 'noisy' spectrum (Fig. 4b). These

spectra are used to determine the elastic constants by the nonlinear least-square optimization method discussed previously. The spectral data from 5 to 10 MHz for normal incidence and from 3 to 12 MHz for oblique incidence is used for reconstruction which corresponds to the procedure utilized in the actual experiment.

Due to the introduced noise the reconstructed set of the polymer film properties is not exactly equal to the original set. It is compared to the original set to study the effects of the noise level and initial guesses. For each noise level and initial guess the procedure is repeated 400 times, the reconstructed parameters (dimensional and nondimensional) are normalized to the original value and average and standard deviation are calculated for the normalized values.

As an illustration, the results of $(\lambda + 2\mu)$ reconstruction from synthetic ‘noisy’ spectra are presented as histogram in Figure 5. Each vertical line represents a number of recon-

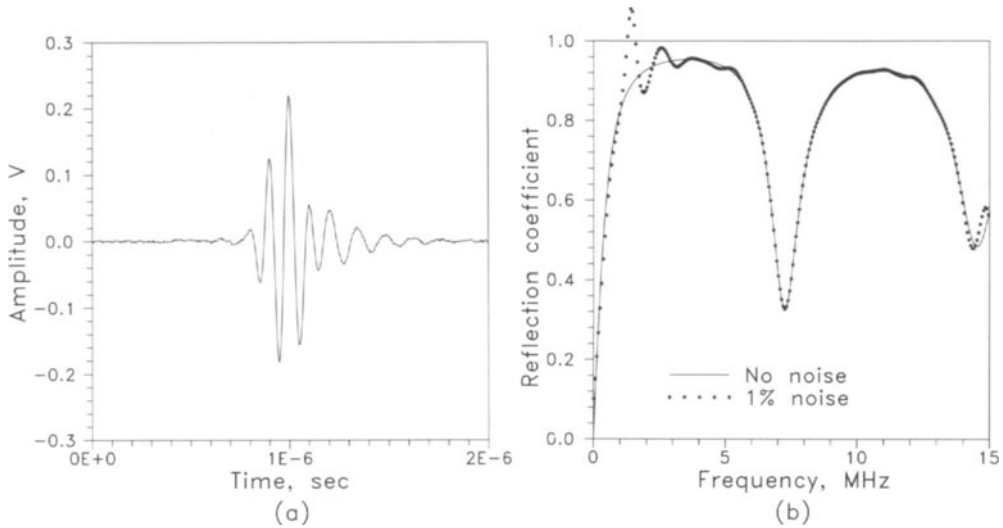


Figure 4. a) A typical synthetic time-domain signal with 1% noise introduced and b) corresponding spectrum.

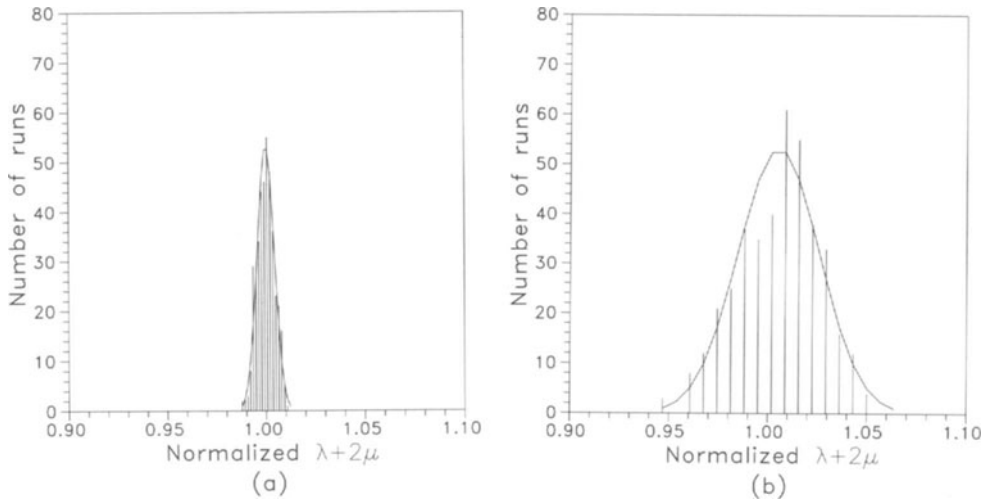


Figure 5. The results of $(\lambda + 2\mu)$ reconstruction from a synthetic ‘noisy’ spectrum with a) 1% noise and b) 5% noise.

Table 1. Average values and standard deviations of the normalized parameter reconstructed from simulated ‘noisy’ spectra with 1% and 5% noise levels. Angle of oblique incidence is 17° . Original values of the dimensional parameters are: $(\lambda + 2\mu)^0 = 4.464$ GPa, $\mu^0 = 0.841$ GPa, $\rho^0 = 1.054$ g/cm³, $h = 0.142$ mm, $V_\ell = 2.058$ km/sec, $V_t = 0.893$ km/sec, $\alpha_\ell^0 = 0.040$, $\alpha_t^0 = 0.063$.

Noise	1%	5%
Z_N/Z_N^0 (ϵ_{Z_N})	1.000 (0.4%)	1.001 (1.9%)
$\bar{h}_\ell/\bar{h}_\ell^0$ ($\epsilon_{\bar{h}_\ell}$)	1.000 (0.0%)	1.000 (0.2%)
$\bar{h}_{\theta\ell}/\bar{h}_{\theta\ell}^0$ ($\epsilon_{\bar{h}_{\theta\ell}}$)	1.000 (0.1%)	0.999 (0.5%)
$\bar{h}_{\theta t}/\bar{h}_{\theta t}^0$ ($\epsilon_{\bar{h}_{\theta t}}$)	1.000 (0.1%)	1.001 (0.5%)
$(\frac{\lambda+2\mu}{(\lambda+2\mu)^0})$ ($\epsilon_{\lambda+2\mu}$)	1.000 (0.4%)	1.005 (2.1%)
μ/μ^0 (ϵ_μ)	1.000 (0.4%)	1.003 (2.0%)
ρ/ρ^0 (ϵ_ρ)	1.001 (0.7%)	0.998 (3.7%)
h/h^0 (ϵ_h)	1.000 (0.5%)	1.004 (2.3%)
V_ℓ/V_ℓ^0 (ϵ_{V_ℓ})	1.000 (0.5%)	1.004 (2.3%)
V_t/V_t^0 (ϵ_{V_t})	1.000 (0.4%)	1.003 (1.8%)
$\alpha_\ell/\alpha_\ell^0$ (ϵ_{α_ℓ})	1.001 (1.0%)	0.998 (5.4%)
α_t/α_t^0 (ϵ_{α_t})	1.005 (1.3%)	1.016 (6.5%)

structed values in an interval of $\sigma_{(\lambda+2\mu)}/3$; a corresponding normal distribution is shown by a solid line in the interval $(\bar{\lambda} + 2\bar{\mu} - 3\sigma_{\lambda+2\mu}, \bar{\lambda} + 2\bar{\mu} + 3\sigma_{\lambda+2\mu})$.

The effect of the initial guess on the results of reconstruction were studied for two different noise levels. It was shown that for initial guesses within $\pm 20\%$ of the originals, the results of inversion are identical. This indicates that the nonlinear least-square optimization is not affected by the initial guesses and that the unique values for the layer properties are determined.

Table 1 summarizes results of nondimensional and dimensional parameter reconstruction for 1% and 5% noise levels. One can see that the error in nondimensional parameter determination (upper part of the table) is relatively small. The error for Z_N determination is larger than that for \bar{h}_ℓ , $\bar{h}_{\theta\ell}$ and \bar{h}_t (parameters responsible for minima positions) which is due to lower reflection sensitivity to normalized impedance. The error for dimensional parameters (lower part of the Table 1 is several times larger than that for nondimensional parameters due to their recalculation by equations (5)–(8). The source of the error increase is in $(\bar{h}_\ell^2 - \bar{h}_{\theta\ell}^2)^{1/2}$, where \bar{h}_ℓ and $\bar{h}_{\theta\ell}$ are close to each other. In general, the precision of dimensional parameter determination does not exceed a few percent with the largest error for density (3.7%) and attenuations (5.4% and 6.5%) at 5% noise level which is unlikely for actual experimental measurement.

EXPERIMENT

The proposed method was applied for characterization of a thin polystyrene film and a polystyrene layer inside an Al-to-Al joint. The joint was prepared using a thin polystyrene layer as a bonding material. Polystyrene was chosen because its properties were not expected to change significantly due to applied pressure and heat during joint preparation. In addition, the polystyrene film can easily be extracted from the joint and its properties measured directly and compared to the ultrasonic measurement results.

The joint was prepared using 6.4 mm thick aluminum alloy coupons (9×3 cm) and 0.25 mm thick polystyrene film. Prior to bonding the aluminum adherend surfaces were sandblasted and washed with acetone. The coupons were bonded by the polystyrene film: samples were heated in 15 minutes to 170°C , then held at this temperature for 30 minutes (Figure 6) under pressure. Spacers were used to ensure homogeneous thickness of the polystyrene layer. Then, the specimen was slowly cooled down to room temperature and the pressure was released.

After the ultrasonic measurement the film was extracted from the joint by peeling and its properties measured directly. The results of the ultrasonic measurements were also compared to the properties of the thick polystyrene plate exposed to the same heat treatment as during the joint preparation.

Properties of the Polystyrene Film Inside the Joint

The polystyrene film properties inside the prepared Al-to-Al joint were determined using a combination of normal/oblique incidence measurements. The reflection spectra were measured in two different locations (L and C) at 0° and at 17° . Results for location C are shown in Figure 7. Three nondimensional parameters, Z_n , \bar{h}_ℓ and α_ℓ , were determined from the reflection spectra at normal incidence. Three more nondimensional parameters, $\bar{h}_{\theta\ell}$, $\bar{h}_{\theta t}$, and α_ℓ , were determined from the oblique incidence data with Z_n , \bar{h}_ℓ and α_ℓ taken as known. The dimensional parameters (4) were calculated using equations (5)–(8). The solid lines in Figures 7 represent spectra calculated from the parameters determined.

After experiment the polystyrene film was extracted from the joint and its density ρ and thickness h measured directly. To determine $(\lambda + 2\mu)$ a normal incidence measurement was performed and the position of the first spectral minimum f_{min} determined. The modulus is then calculated from: $\lambda + 2\mu = 4h^2 \cdot \rho \cdot f_{min}^2$.

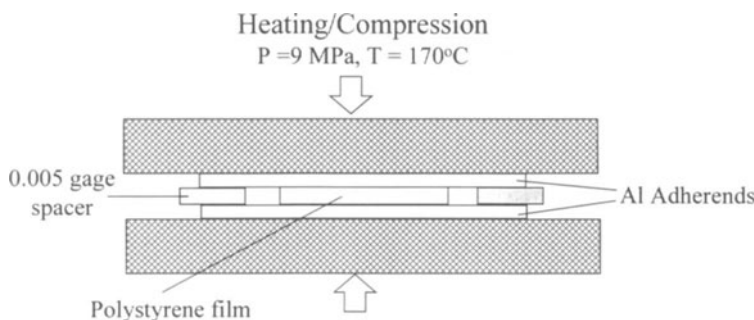


Figure 6. A schematic of the Al/polystyrene/Al joint preparation.

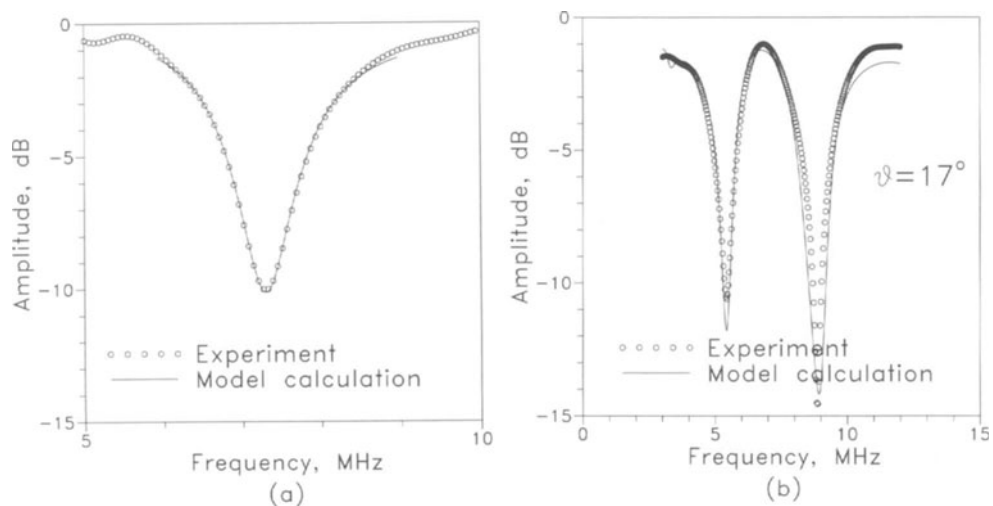


Figure 7. Reflection spectra of the polystyrene layer inside the Al-to-Al joint measured at a) 0° and b) 17° . Model calculations (solid lines) are done in the frequency range used for inversion.

Table 2. Comparison of elastic properties of the film polystyrene in the joint, removed from the joint and heat treated bulk polystyrene.

		$\lambda + 2\mu$, GPa	μ , GPa	ρ , g/cm ³	h , mm	α_ℓ	α_t
Polystyrene film in the joint	Point L	4.43	0.833	1.06	0.141	0.040	0.063
	Point C	4.50	0.860	1.08	0.140	0.038	0.045
Polystyrene film removed from joint	Point L	4.54		1.072*	0.143*		
	Point C	4.41		1.072*	0.145*		
Heat treated polystyrene		4.69	.828	1.072*			
* — direct measurement							

Table 2 compares the film properties determined ultrasonically inside the joint to the extracted film properties and to the heat treated thick polystyrene properties (measured by SRBW method). The data measured directly (i.e. thickness measured by micrometer and density by a water displacement method) is marked by an asterisk (*). Relative error for direct density measurement is estimated to be 0.3% (± 0.003 g/cm³), for thickness - 2% (± 3 μ m). This results in more than 4% error in the ($\lambda + 2\mu$) for the film extracted from the joint (or ± 0.2 GPa). An additional source of error is in nonhomogeneous thickness of the layer: up to 5 μ m difference in the ultrasonic beam diameter circle of ≈ 4 mm. Taking this into account the ($\lambda + 2\mu$) values measured inside and outside the joint are in good agreement. The shear modulus values are reasonably close to those of the heat treated bulk polystyrene. The film thickness and density are in good agreement with the directly measured values. The properties measured for the two points (L and C) are close to each other with the difference attributed to variation of properties from point to point and to experimental error.

CONCLUSIONS

This paper describes an ultrasonic method for property determination for an isotropic layer embedded between two known materials (similar or dissimilar). The method allows simultaneous determination of all the layer properties: thickness, density and elastic moduli from normal and oblique incidence reflection spectra. It describes reflection from the layer as a function of four nondimensional parameters and longitudinal and shear wave attenuations. The algorithm for the nondimensional parameter determination from the experimentally measured reflection spectra at normal and oblique incidence (at one angle only) is developed. The thickness, density and elastic moduli of the layer are calculated from the nondimensional parameters determined. The method stability against the experimental noise is studied and the algorithm is accordingly optimized. The method is validated experimentally using thermoplastic joints.

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