DETERMINATION OF ELASTIC CONSTANTS BY LFB ACOUSTIC MICROSCOPE

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ABSTRACT

A new method of measuring elastic constants of bulk solid materials by means of the line-focus-beam (LFB) acoustic microscope has been developed. An LFB acoustic microscope has been applied to precisely measure the velocity of leaky surface acoustic waves (LSAWs) propagating on water-loaded specimens. The LSAW velocity is directly related to the elastic properties of specimens, so that it is possible to determine the elastic constants by theoretical analysis. For this study, an algorithm has been devised that can calculate the SAW velocity for a free-surface-conditioned specimen using the measured LSAW velocity. Elastic constants have been determined by computer-fitting of the SAW velocity in numerical calculations. In this method, precise measurements of the longitudinal velocity and the density are also carried out for the same specimen, in order to reduce the number of unknown parameters. Experiments are demonstrated at a VHF range for isotropic samples, such as fused quartz (SiO₂) and some kinds of borosilicate glasses. Two independent components of stiffness constants, $C_{1,1}$ and $C_{4,4},$ have been determined with high accuracy and compared with the published technical data.

1. Introduction

In recent studies on the LFB acoustic microscope, demonstrational experiments have been extensively done to show its possible applications to material analyses in the research fields of material science and nondestructive evaluation, for example, acoustic anisotropy, acoustic inhomogeneity, structural analysis of polycrystalline materials, film thickness measurement, and viscoelastic analysis of dental materials [1-5]. Another expectable application presented here is for determining material constants.

For determination of elastic constants, it is conventional to make velocity measurements of both longitudinal and shear waves by various kinds of ultrasonic methods including the optical diffraction method [6]. The ultrasonic transducers, made of piezoelectric plates at lower frequencies or ZnO thin films at higher frequencies, must be usually fabricated on one end of specimens, of which both end surfaces are polished with parallelism. On the other hand, the method by the LFB acoustic microscope has the great advantage that nondesructive and noncontacting measurements can be made without fabrication of any ultrasonic transducers for anisotropic as well as isotropic materials.

In this paper, a new method is developed theoretically and experimentally, taking the simplest case of isotropic materials.

2. Method

The method is discussed taking isotropic materials for simplicity as shown in Fig. 1.

2.1. LSAW mode

First, the propagation characteristics of LSAW mode, corresponding to the sample configuration with the water loading on specimens, should be considered for this study.

Under the assumption that acoustic loss in specimens is negligible, the characteristics equation is given by [7]

$$4\beta_{1}\beta_{2} - (1+\beta_{1}^{2})^{2} = j(\rho_{w}\beta_{2}/\rho\beta_{3})(1-\beta_{1}^{2})^{2}, \quad (1)$$

where

$$\beta_{1} = (1 - (k_{t}/k)^{2})^{1/2},$$

$$\beta_{2} = (1 - (k_{1}/k)^{2})^{1/2},$$

$$\beta_{3} = ((k_{u}/k)^{2} - 1)^{1/2},$$
(2)



ISOTROPIC SOLID

Fig. 1. Schematic sample configuration for propagation of LSAW and SAW modes.

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and the wavenumbers are defined as

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$$k_t = \omega / V_t$$
, $k_l = \omega / V_l$, $k_w = \omega / V_w$. (3)

 V_1 and V_t are the velocities of longitudinal and shear waves in a specimen, respectively, and V_u is the longitudinal velocity of water. ω is the angular frequency. ρ and ρ_u are the densities of specimen and water, respectively. The velocities are related to the stiffness constants C_{ij} (or Lame's constant λ) and the densities as

$$V_{t} = (C_{44}/\rho)^{1/2},$$

$$V_{1} = (C_{11}/\rho)^{1/2},$$

$$V_{w} = (\lambda/\rho_{w})^{1/2}.$$
 (4)

In Eq.(1), the complex wavenumber $k_{1,s,a,w}$ that is the solution of k for LSAW mode is defined as

$$k_{1saw} = (\omega/V_{1saw})(1+j\alpha_{1saw}) , \qquad (5)$$

where V_{1 saw} and $\alpha_{1 saw}$ are the velocity and the normalized attenuation factor, respectively. The characteristic equation given in Eq. (1) can not be analytically solved, so that the solution should be obtained by numerical calculations.

2.2. Principle

In the quantitative material characterization method by means of the LFB acoustic microscope, coupling water acts as a reference liquid in measurements because the physical properties of water. V and $\rho_{\rm w}$, are exactly known. As the V_{1 saw} and $\alpha_{1 saw}$ can be measured with the LFB acoustic microscope, the unknown parameters are C₁₁, C₄₄, and ρ in Eq. (1). It is impossible, however, to determine all the three unknown parameters can be determined by computer-fitting of the measured data in numerical calculations for LSAW mode with Eq. (1), if we measure either the density or the longitudinal velocity. In this method, there should be a serious problem on the accuracy with relatively large error of several percent. This is mainly because it is very difficult to measure the $\alpha_{1 saw}$ accurately in comparison with the V_{1 saw}.

To raise the measurement accuracy, it is necessary to cultivate the method without use of the measured $\alpha_{]saw}$. This suggests us to use the propagation characteristics for SAW mode. Therefore, the key point is how to evaluate the SAW velocity properly from the measured LSAW velocity which will be described later in detail.

The characteristic equation giving the solution of $k=k_{aw} = \omega/V_{aw}$ (V ; the SAW velocity) for SAW mode is simply represented for a free-space condition on the surface of a specimen as follows:

$$4\beta_{1}\beta_{2}^{-}(1+\beta_{1}^{2})^{2}=0.$$
 (6)

To determine the elastic constants by using the

above equation, the number of unknown parameters should be one. We can obtain the density ρ and C_{11} from the equation that $C_{11} = \rho V_1^{-2}$ by the measurements of the density and longitudinal velocity. So, it is possible to determine another stiffness constant C_{44} for the isotropic case.

2.3. Water loading effect

Using the perturbation method, let us consider the water loading effect to derive the approximate relation between the velocities of V_{1saw} and V_{saw} . We can define k_{1saw} as follows:

$$k_{1saw} = k_{saw} + \Delta k$$
, (7)

where Δk is the complex number and $|\Delta k/k|$ is much smaller than unity. Using Eqs. (1), (6), and (7), we obtain the Δk to be represented by

$$\Delta k = (B^{2}C/(A^{2}+B^{2}C^{2}))k_{saw} + j(AB/(A^{2}+B^{2}C^{2}))k_{saw},$$
(8)

where

$$A = (1 + \beta_1^2)^2 (2 + 1/\beta_1^2 + 1/\beta_2^2 - 8/(1 + \beta_1^2))$$

$$B = (\rho_w / \rho) (\beta_2 / \beta_3) (1 - \beta_1^2)^2$$

$$C = 1/\beta_2^2 + 1/\beta_3^2 .$$
(9)

Eliminating Δk with Eqs. (7) and (8), and using Eq. (5) and $k_{saw} = \omega/V_{saw}$, the following approximate relations are obtained

$$V_{1saw}/V_{saw} = (A^2 + B^2 C^2)/(A^2 + B^2 C^2 - B^2 C)$$
, (10)

$$\alpha_{1saw} = AB/(A^2 + B^2 C^2 - B^2 C) .$$
 (11)

It can be seen from Eq.(10) that $V_{1saw}/V > 1$. This means that the water loading effect gives rise to the increase in the velocity.

2.4. Algorithm

As the right hand side term in Eq. (10) includes the unknown parameters of C_{44} and V_{saw} , the value of V_{saw} can not be either obtained directly from the Eq. (10). We now consider the following approximation with the measured value of V_{1saw} . As the change in SAW velocity due to the water loading on a specimen is very small, the value of V_{saw} is nearly equal to that of V_{1saw} . So, we can use the measured V_{1saw} as the initial value V_{saw} . Substituting the V_{saw} into the V_{saw} in Eq. (6) with the precisely measured C_{11} and P, the approximate value of C_{44} can be numerically calculated. As in the calculation the water loading effect is neglected, the accuracy is still poor.

To get a more accurate value, let us introduce the following approximation that the change in SAW velocity due to the water loading effect with the approximate value C_{44} ' calculated above is approximately equal to that with the true value

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 C_{44} , that is,

$$V_{1saw}'/V_{saw}' = V_{1saw}/V_{saw}$$
(12)

The V₁ saw ' in Eq. (12) can be calculated using Eq. (1) with C_{44} ', where the water-loading effect is approximately taken into consideration. From Eq. (12), a more accurate velocity V_{5aw}'' can be obtained as

$$V_{saw}'' = V_{saw} = V_{1saw}(V_{saw}'/V_{1saw}'), \quad (13)$$

Substituting V '' into the Eq. (6), a more accurate C_{44} ''saw be determined. The abovementioned procedure are repeatedly carried out until an ultimate value of C_{44} can be obtained. A flow chart of the numerical calculations to determine the elastic constants is summarized in Fig. 2.

3. Experiments

In this elastic-constant determination method, three quantities, such as the LSAW velocity, the longitudinal velocity, and the density, should be measured.

The LSAW velocity can be measured by means of the LFB acoustic microscope. A block diagram of the recent system [3] is shown in Fig. 3. Fig. 4 is the cross-sectional geometry of the LFB acoustic lens to show basic concept of V(z) curve measurements. LSAWs are excited on the boundary between a specimen and water at the critical angle of θ_{1} saws the LSAW velocity can be precisely determined. A detailed description of the system and measurement principle has been made in the literature [1]. Experiments are carried out using an acoustic LFB sapphire lens of 1.0mm in radius at a frequency of 225 MHz.

Longitudinal velocity measurements can be made by the pulse interference method. The principle is shown in Fig. 5. A plane-beam acoustic device consisting of ZnO-film transducer on SiO₂ buffer rod is incorporated in the LFB acoustic system by replacing the LFB acoustic device. Careful alignment is made to get parallelism between two surfaces of SiO₂ buffer rod and specimen. An RF pulse is transmitted through a SiO_2 buffer rod and water on a specimen with a thickness h. Partial reflections and transmissions at each interface occur. Among pulse echoes received by the transducer, we take note of the two echoes, namely, P1 and P2, in Fig. 5. P1 is directly reflected from the top surface of the specimen, while P2 from the bottom surface. We adjust the length of RF pulse long enough that the two echoes may overlap and interfere. The interfered signal is gated out and measured by sweeping the frequency. A series of interference maxima and minima can be observed. The frequency periodicity Δf is related to the longitudinal velocity V_1 and the thickness h as

$$\Delta f = V_1 / 2h . \tag{14}$$



Fig. 2. A flow chart of elastic-constant determination.



Fig. 3. Block diagram of the LFB acoustic microscope system.

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Fig. 4. Cross-sectional geometry of LFB acoustic lens to show V(z) curve measurements.



Fig. 6. V(z) curve measured for fused quartz at 225 MHz.

Therefore, we can determine V_1 using measured Δf and h. Measurements of the thickness are made by the digital micrometer, of which the accuracy is better than $\pm 1 \mu m$. The density is measured by the Archimedes' method.

Experiments are demonstrated for isotropic samples; two fused quartz and two kinds of borosilicate glasses with both surfaces optically polished and sufficient parallelism. Fig. 6 shows the typical V(z) curve measured for a fused quartz sample named as FQ1. From this V(z) curve, the velocity V was determined to be 3437 m/s. Fig. 7 shows the typical frequency response of the interference output measured for the same sample. From this waveform, we can measure $\Delta f=1.7339$ MHz. Using Eq. (14) with the measured thickness of 1.716 mm, the longitudinal velocity was determined to be 5951 m/s. Using a set of data, that is, V₁, V₁saw, and ρ , final determination is made for two independent stiffness constants, C₁₁ and C₄₄, for isotropiz materials. C₁₁ is determined to be 7.800x10 N/m



Fig. 5. Experimental arrangement of longitudinal velocity measurements by the pulse interference method.



Fig. 7. Frequency response of interference output for fused quartz in longitudinal velocity measurements by the pulse interference method.

directly from the equation that $C_{11} \approx \rho V_1^2$. On the other hand, C_{44} is determined to be 3.147×10 N/m², according to the analysis procedure.

Experimental data obtained for other specimens are summarized in Table 1. The determined material constants are presented together with the published data in Table 2. In the present measurements, the accuracy of C_{11} is several parts in 10 mainly dominated by that of the thickness measurements, while the accuracy of C_{44} is estimated to be better than one part in 10 dominated by that of the LSAW velocity measurements. For the density measurements, the accuracy was a few parts in 10⁴. From the results, it can be said that the values measured here are in good agreement with the published values within a maximum difference of one or two percent. It is very exciting that, between two SiO₂ specimens supplied from different companies, slight differences in the values were detected, which might be due to some changes of mechanical properties in them.

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| Sample | ρ (10 ³ kg/m ³) | V _{lsaw} (m/s) | V _l (m/s) | h (mm) | ∆f (MHz) |
|--------|--|-------------------------|----------------------|--------|----------|
| FQ1 | 2.203 | 3437 | 5951 | 1.716 | 1.7339 |
| FQ2 | 2.201 | 3437 | 5932 | 1.922 | 1.4889 |
| 7740 | 2.226 | 3145 | 5560 | 2.003 | 1.3879 |
| E6 | 2.185 | 3061 | 5405 | 2.571 | 1.0511 |

Table 1. Measured values.

: Fused quartz (No.T4040) produced by Toshiba Ceramics Co.,Ltd. : Fused quartz (No.ES) produced by Japan Quartz Glass Co.,Ltd. F01

FQ2

7740 : Pyrex glass produced by Corning Co., Ltd. E6

: Borosilicate glass produced by Ohara Optical Mfg.Co.,Ltd.

Table 2. Determined values compared with published data.

| Sample | C ₁₁ (10 ¹⁰ N/m ²) | C ₄₄ (10 ¹⁰ N/m ²) | ρ (10 ³ kg/m ³) | Ref. |
|--------|--|--|--|------|
| FQ1 | 7.800 (7.85) | 3.147 (3.12) | 2.203 (2.2) | a |
| FQ2 | 7.746 (7.85) | 3.149 (3.12) | 2.201 (2.2) | a |
| 7740 | 6.881 (6.97) | 2.626 (2.62) | 2.226 (2.23) | b |
| E6 | 6.382 (6.33) | 2.440 (2.40) | 2.185 (2.18) | с |

Published data in parentheses.
a) W.P.Mason, Physical Acousitcs and the Properties of Solids (McGraw-Hill, New York, 1958), p.17.
b) Technical data from Corning Co.,Ltd.
c) Technical data from Ohara Optical Mfg.Co.,Ltd.

4. Conclusion

A new method of determining elastic constants of solid materials using the LFB acoustic microscope has been developed. The demonstration has been successfully made for isotropic materials. The method can be easily expanded for the case of anisotropic materials because the procedure is the quite same as described here for an isotropic case. In anisotropic materials, there are a number of unknown parameters depending on crystal systems, so that the more complicated characteristic equations to solve SAW and LSAW modes should be employed in numerical calculations. This will be one of the most promising applications because measurements can be simply and accurately made for anisotropic materials without fabrication of any ultrasonic transducers on specimens. It can be also expected, in the near future, that the development in this application will lead to determination of elastic constants for as-grown thin films as well as implanted and diffused layers used in electronic devices, as suggested in the literatures [1,8].

Acknowledgments

The authors are very grateful to H. Takahashi for his helpful assistance in density measurements. This work was supported in part by the Research Grant-in-Aids for the Japan Ministry of Education, Science & Culture.

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