Measurement of Anisotropic Elastic Moduli in Local Area by Acoustic Microscope* (The Case of Cubic Single Crystal)

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In this paper, a new method is suggested for the nondestructive measurement of elastic moduli on the local surface of an anisotropic material by an amplitude and phase acoustic microscope. It is confirmed that the anisotropic wave velocities in a local area are determined from the complex $V(z)$ curve measured by a line-focus acoustic lens. The experimental analyses were carried out for single crystal of cubic system, (001) face of gallium arsenide, with water or mercury as the coupler. The velocity of the surface wave was clearly seen when the acoustic impedance of the coupler was smaller than that of the specimen. The velocity of the longitudinal wave was successfully measured, provided the acoustic impedance of the coupler was comparable to that of the specimen. Then, the elastic moduli in a single crystal of gallium arsenide was determined from the measured longitudinal and surface wave velocities according to elastodynamic theory. It was shown that the present method is very useful in determining the anisotropic elastic moduli of a cubic system in a local area.

Key Words: Nondestructive Inspection, Supersonic Wave, Anisotropy, Experimental Mechanics, Acoustic Microscope, Elastic Modulus, Complex $V(z)$ Curve, Reflectance Function, Ultrasonics

1. Introduction

In recent years, nondestructive method to measure mechanical properties of solid material in a local area has been desired in order to control quality of micro-electron devices and to estimate the properties of the inhomogeneous materials, such as advanced

composite materials and functionally gradient materials, against a background of miniaturizing machine elements and parts. The elastic modulus, one of the fundamental mechanical properties, has been widely measured by several kinds of static test methods⁽¹⁾ and dynamic test methods⁽²⁾ for the objective area over 1 mm^2 . For a local area under 1 mm^2 , it will be difficult for these conventional methods to measure elastic moduli nondestructively.

Acoustic microscope has attracted the attention of some researcher as the tool to obtain nondestructively the mechanical properties and stress state of solid material from the acoustic wave velocities measured in a local area under 1 mm^2 ^{(3),(4)}. In general, the velocity of leaky surface wave for a specimen is obtained by the periodicity in the amplitude variation of $V(z)$ curve^{(5),(6)}, that is defined as the amplitude of reflected wave from the surface of the specimen as a function of z direction normal to the surface of the specimen. The leaky surface wave is affected by both properties of a solid specimen and a coupler which is an acoustic medium setting between an acoustic lens and a solid specimen. In the field of acoustic microscopy, many researchers have been explored the every

* Received 3rd April, 1998. Japanese original: Trans. Jpn. Soc. Mech. Eng., Vol. 63, No. 611, A (1997), p. 1467-1474 (Received 1st October, 1996)

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possibility to measure the distribution of residual stress^{(7),(8)} and the elastic moduli^{(9)–(11)}, and to determine the area of plastic deformation^{(12),(13)} and the anisotropy⁽¹⁴⁾ and so on by using $V(z)$ curve method.

Liang et al.⁽¹⁵⁾ showed theoretically and experimentally that the reflectance function at the coupler-specimen interface can be obtained from the measurement and analysis of the complex $V(z)$ curve including both the amplitude and the phase of reflected wave from the surface of the specimen. Endo et al.⁽¹⁶⁾ discussed the possibility to measure the velocities of three acoustic waves, that is to say longitudinal wave, transverse wave and Rayleigh wave, in local areas of 100–400 μm in diameter, using higher frequencies than 100 MHz by complex $V(z)$ curve method. Nishida et al.⁽¹⁷⁾ applied to the nondestructive measurements of the elastic moduli in local area for homogeneous and isotropic solid materials using the velocities of the longitudinal wave and transverse wave determined by complex $V(z)$ curve method. Actually, general polycrystalline materials show the preferred orientation in local area, such as the texture. Thin solid films on single crystal behave as the anisotropic materials by epitaxial growth. Then, the new method has been required to measure accurately the elastic moduli of the anisotropic materials⁽¹⁸⁾.

The point-focus lens and line-focus lens have been used for the acoustic microscope. The spherical wave radiated from the point-focus lens into coupler generates the surface waves in all directions on a surface of specimen. Then, the result measured by the point-focus lens give the average value for the azimuthal properties. On the other hand, the cylindrical wave radiated from the line-focus lens into the coupler generates the surface wave in one direction on a surface of specimen. Then, it is possible to measure the azimuthal characteristic of the anisotropic materials using the line-focus lens⁽⁶⁾. A large number of studies have mainly been made on the measurement of the leaky surface wave velocities for anisotropic materials by $V(z)$ curve method using the line-focus lens.

In the present paper, the complex $V(z)$ curve method is expanded into the measurement of elastic anisotropy using the line-focus lens. Because the information about not only the critical angle of the acoustic wave but also the kind of the acoustic wave is obtained by this method, it is possible to measure the several wave velocities for the anisotropic materials simultaneously. From these velocities, the anisotropic elastic moduli in a local area on a single crystal of cubic system, that has many symmetry axes, can be calculated, based on the theoretical relation among wave velocities, elastic moduli and

Fig. 1 Definition of coordinate used in analysis for line-focus lens

density.

2. Complex $V(z)$ Curve Method for Line-focus Lens

2.1 Theory of complex $V(z)$ curve for line-focus lens

After due consideration of the symmetry of the shape on the tip of line-focus lens, the origin of the coordinate system (x, y, z) is placed on the center of the focal plane, as shown in Fig. 1. For the line-focus lens, the formulation of the complex $V(z)$ curve can be expressed as the following function on x - z plane^{(19),(20)}.

$$V(z) = \int_{-\infty}^{\infty} P^2(x/f) R(x/f) \times \exp[j2k_L^L z \{1 - (x^2/f^2)\}^{1/2}] dx, \quad (1)$$

where R, P, f and k_L^L are the reflectance function between a coupler and a specimen, the pupil function of the acoustic lens, the focal length of the acoustic lens and the wave number of the longitudinal wave in the coupler, respectively. $j = \sqrt{-1}$.

If we define

$$\sin \theta = x/f \quad (2)$$

$$u = k_L^L z \quad (3)$$

$$\Theta = \cos \theta. \quad (4)$$

Equation (1) can be rewritten as follows:

$$V(u) = \int_{\cos \theta_m}^1 \hat{P}^2(\Theta) R(\Theta) \exp[ju\Theta] d\Theta, \quad (5)$$

here θ_m is a half-aperture angle of the acoustic lens and

$$\hat{P}^2(\Theta) \equiv P^2(\Theta) \frac{f \cdot \Theta}{\sqrt{1 - \Theta^2}}. \quad (6)$$

Equation (5) represents that the multiplication of the transfer function $\hat{P}^2$ and the reflectance function R is the Fourier transform of complex $V(z)$ curve with respect to Θ . In other words, the information of the reflectance function can be obtained from the Fourier transform of complex $V(z)$ curve, though the Fourier transform of complex $V(z)$ curve includes the information of the transfer function. When the

transfer function varies steadily with respect with θ , that is to say normalized wave number k_{Lz}^f/k_L^f , $\hat{P}^2 R$ can exhibit the signals of critical angles on R . Here k_{Lz}^f means the z axial component of wave number in the coupler k_L^f . Therefore, the wave velocities can be obtained from the critical angle with Snell's law⁽¹⁶⁾.

2.2 Verification of theory

In order to verify the complex $V(z)$ curve method applied to the line-focus lens, the wave velocities of homogeneous and isotropic materials are measured by the point-focus lens and the line-focus lens. The used apparatus is the amplitude and phase acoustic microscope (Olympus Optical Co., Ltd.). The measured transfer function is shown in Fig. 2, where distilled water is used as a couple. The results by line-focus lens and point-focus lens indicate solid line and dotted line in Fig. 2, respectively. The relative intensities of two lenses are roughly similar each other. Two relative intensities have the maximum value in the vicinity of normalized wave number $k_{Lz}^f/k_L^f=1$, that corresponds to the center axis of lens, and gradually decline as the normalized wave number decrease, because the acoustic wave excited by the transducer is strengthened at the center axis of lens. Furthermore, two phases of transfer functions remain constant to approximately 0 deg. for all normalized wave number, because the propagation length of acoustic wave in the acoustic lens is longer than the wave length of the acoustic wave. Thus, the wave velocities can be obtained from the Fourier transform of measured complex $V(z)$ curve.

Next, the wave velocities measured by the complex $V(z)$ curve method for the line-focus lens are

Fig. 2 Transfer function measured with water coupler (400 MHz), solid line: line-focus lens, dotted line: point-focus lens

compared with those for the point-focus lens. The specimens are two kinds of glasses (HOYA corp.), defined as type **a** and type **b**, and fused quartz. These wave velocities are different from each other, as shown in Table 1. The specimens are amorphous, that is homogeneous and isotropic. Water and mercury^{(25)–(27)} in Table 1 were used as a coupler. The measured areas are a circle of about 0.3 mm in diameter for the point-focus lens and a rectangle of about 0.3 mm length and 0.8 mm width for the line-focus lens, respectively.

Figure 3 shows the Fourier transform of complex $V(z)$ curve measured for glass type **b** with water coupler. In this case, the normalized wave numbers for the critical angles of Rayleigh wave in the phase, ∇C , are approximately same in the results of two lenses, though the values of the relative intensities are different from each other.

For two kinds of coupler and three kinds of specimen, Table 2 shows the wave velocities determined from the measured complex $V(z)$ curve and Snell's law. For the measurement of once a combination of a specimen and a coupler, the results by the

Table 1 Reference data of samples

Sample	Velocity		Density [kg/m ³]
	C_L [m/s]	C_T [m/s]	
Glass a ⁽²¹⁾	5330	2987	3.58
Glass b ⁽²¹⁾	5170	2905	3.62
Fused quartz ⁽²²⁾	5953	3757	2.2
Water ⁽²³⁾	1500	—	1.0
Mercury ⁽²⁴⁾	1450	—	13.6

C_L : Longitudinal wave, C_T : Transverse wave

Fig. 3 Fourier transform of complex $V(z)$ curve measured for water/glass type **b** (400 MHz), solid line: line-focus lens, dotted line: point-focus lens, ∇C : Rayleigh wave

Table 2 Measured wave velocities of glass and fused quartz by point-focus lens and line-focus lens

Specimen	Glass type a			Glass type b			Fused quartz		
	Mercury		Water	Mercury		Water	Mercury		Water
Wave	C_L [m/s]	C_T [m/s]	C_R [m/s]	C_L [m/s]	C_T [m/s]	C_R [m/s]	C_L [m/s]	C_T [m/s]	C_R [m/s]
Point-focus lens	5091	2964	2745	4940	2872	2680	5812	3677	3418
Line-focus lens	5080	2945	2739	4940	2851	2668	5813	3692	3405

C_L : Longitudinal wave, C_T : Transverse wave, C_R : Rayleigh wave

point-focus lens agree with those by the line-focus lens. Consequently, the wave velocities can be accurately measured by the complex $V(z)$ curve method expanded into the line-focus lens.

3. Measurements of Wave Velocities for Anisotropic Materials

3.1 Theoretical reflectance function for anisotropic materials

The (001) face of gallium arsenide, that is classified into single crystal of cubic system, was employed, in order to clear the characteristic of the method by using the line-focus lens. The single crystal of cubic system has only three independent elastic moduli. The matrix of elastic modulus can be expressed as follows:

$$\begin{bmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{bmatrix} \quad (7)$$

Nishida et al.⁽²⁸⁾ showed that the signals of critical angles for longitudinal wave and transverse wave measured by the point-focus lens could be recognized clearly by selecting the suitable coupler, considering that the reflectance function is decided by both acoustic impedances of the coupler and the specimen. Then, in order to detect clearly the signals of critical angle for the acoustic waves by the complex $V(z)$ curve method using the line-focus lens, the mercury is also employed as a coupler, in addition to water that is fundamentally used as a coupler.

Figure 4 shows the theoretical reflectance function on the (001) face of gallium arsenide at the azimuthal angle of 25 deg. from [100] direction^{(29),(30)}. The material characteristics of water, mercury and gallium arsenide in the computation are shown in Table 1 and 3⁽³¹⁾. The solid line and dotted line denote the results of mercury coupler and water coupler, respectively. When water is used as a coupler, the relative intensity of the reflectance function is equal to 1 and the phase of that is equal to 0 at the normalized wave number coincided with the critical angle of

Fig. 4 Theoretical reflectance function on (001) face of gallium arsenide, solid line: mercury, dotted line: water, ($\phi=25$ [deg.] from [100] direction), ∇ : critical angle in mercury coupler, ∇ : water coupler, A: longitudinal wave, C: Rayleigh wave, D: pseudo Rayleigh wave

longitudinal wave ($k_{Lz}^f/k_L^i=0.9553$, ∇A) where the total reflection of acoustic wave occurs. At the normalized wave numbers of Rayleigh wave ($k_{Lz}^f/k_L^i=0.8385$, ∇C) and pseudo Rayleigh wave ($k_{Lz}^f/k_L^i=0.8498$, ∇D), the imaginary part of reflectance function is equal to 0 and the relative intensity and the phase vary extremely and complexly. However, at the normalized wave number of transverse wave, the relative intensity and the phase do not vary remarkably⁽³⁰⁾. The single for the critical angle of longitudinal wave measured by using a coupler of mercury become more clear compared with a coupler of water, as shown by ∇A in Fig. 4. The phase measured by using mercury also deviates from 0 deg. at the normalized wave number except near the critical angle of Rayleigh wave and pseudo Rayleigh wave.

3.2 Measurements of reflectance function for anisotropic materials

Figure 5 and 6 show the Fourier transform of the complex $V(z)$ curves measured by using water and mercury as a coupler for the (001) face of gallium arsenide, respectively. In order to detect the signal of the critical angle for acoustic wave clearly, the desirable frequencies were selected: 400 MHz for water coupler and 450 MHz for mercury coupler. The area measured for the (001) face of gallium arsenide by

Fig. 5 Fourier transform of complex $V(z)$ curve measured for water/(001) face of gallium arsenide, 400 MHz, $\phi=25.5$ [deg.] from [100] direction, ∇C : Rayleigh wave, ∇D : pseudo Rayleigh wave

Fig. 6 Fourier transform of complex $V(z)$ curve measured for mercury/(001) face of gallium arsenide, 450 MHz, $\phi=27$ [deg.] from [100] direction, ∇A : longitudinal wave

using line-focus lens is a rectangle of approximate 0.5 mm length and 0.8 mm width. In this figure, ϕ is the azimuthal angle of single crystal from the [100] direction.

The signals of critical angles of Rayleigh wave (∇C) and pseudo Rayleigh wave (∇D) can be recognized on the phase variation of the Fourier transform of the measured complex $V(z)$ curve in the case of water coupler as shown in Fig. 5. However, it is difficult for the signal of critical angle for longitudinal wave to be recognized on the relative intensity and the phase of the Fourier transform of measured complex $V(z)$ curve. When the acoustic impedance of a coupler is smaller than that of longitudinal wave for specimen, the phase in the Fourier transform of the complex $V(z)$ curve changes more rapidly at the critical angles for acoustic waves.

The signal of the critical angle for the longitudinal wave (∇A) can be recognized in the relative intensity in the case of mercury coupler as shown in Fig. 6. When the acoustic impedance of a coupler is comparable to that of the longitudinal wave of specimen, the signal of relative intensity could be measured much clearly at the experiment. At a range of $0.95 > k_{Lz}^f/k_L^f > 0.90$, the phase obtained by the experiment is different from that obtained by the theory as shown by solid line in Fig. 4. It is inferred for this reason that the relative intensity at this range is smaller than that at the normalized wave number except this range. In the future, this reason should be considered in detail.

Therefore, when the coupler is selected prudently so that the acoustic impedance of coupler is comparable to that of the specimen, the clear measurements of signals for the acoustic waves could be achieved. The experimental results with the line-focus lens agreed with those with the point-focus lens in respect of the function of the coupler⁽²⁸⁾.

3.3 Measurements of acoustic wave velocities

There are the symmetry axes of single crystal at the intervals of 45 deg. like [100] direction or [110] direction on (001) face of gallium arsenide employed as a specimen. The longitudinal wave, transverse wave and Rayleigh wave have the maximum value or minimum value of each velocities at these direction. Then, based on the direction that is conjectured as the [100] direction of single crystal for specimen, the complex $V(z)$ curves are measured by the line-focus lens at every 3 deg. from -9 deg. to +54 deg. According to Snell's law, the variation of wave velocities to the azimuthal angle of single crystal is obtained from the measured critical angle. In the case of using mercury as a coupler, the relationship between the azimuthal angle of single crystal ϕ and the velocity of the longitudinal wave is shown in Fig. 7. In the case

Fig. 7 Measured velocities of longitudinal wave for (001) face of gallium arsenide, mercury coupler, 450 MHz, $\phi=0$ [deg.]: [100] direction, $\phi=90$ [deg.]: [110] direction

Fig. 8 Measured velocities of surface wave (Rayleigh wave and pseudo Rayleigh wave) for (001) face of gallium arsenide, water coupler, 400 MHz, $\phi=0$ [deg.]: [100] direction, $\phi=90$ [deg.]: [110] direction

of using water as a coupler, the relationships between the azimuthal angle of single crystal ϕ and the velocities of pseudo Rayleigh wave and Rayleigh wave are shown in Fig. 8. Here the range from 0 deg. to 45 deg. is expressed into Fig. 7 and Fig. 8, because the longitudinal wave, pseudo Rayleigh wave and Rayleigh wave have the maximum value or minimum value of the velocity at the [100] direction or [110] direction. The experimental results for longitudinal wave, pseudo Rayleigh wave and Rayleigh wave are in fair agreement with the theoretical results^{(4),(32)}, computed from the data in Table 3. Both velocities of pseudo Rayleigh wave and Rayleigh wave could be measured simultaneously at a range of the azimuthal angles from 22.5 deg. to 31.5 deg. Then, it is shown that several kinds of acoustic wave velocities for anisotropic materials could be measured by complex $V(z)$ curve method using the line-focus lens.

4. Calculation of Elastic Moduli

The elastic moduli of single crystal are calculated

Table 3 Elastic moduli and density of gallium arsenide

	Elastic modulus			Density [10^3 kg/m^3]
	c_{11} [GPa]	c_{12} [GPa]	c_{44} [GPa]	
Reference ⁽³⁰⁾	118.8	53.8	59.4	5.31

Table 4 Calculated elastic moduli of gallium arsenide by this method

Elastic modulus	c_{11}	c_{12}	c_{44}
[GPa]	118.1	54.6	60.6

from the velocities of longitudinal wave and surface wave, that is to say pseudo Rayleigh wave and Rayleigh wave, measured in the previous chapter. The measured velocities, $C_L^{(M)}$ and $C_R^{(M)}$, of longitudinal wave and surface wave are compared with theoretical velocities, $C_L(c_{11}, c_{12}, c_{44})$ and $C_R(c_{11}, c_{12}, c_{44})$, obtained by the theoretical equation^{(4),(33)} related among elastic moduli, wave velocities and density of the specimen. The residual function of the wave velocities obtained by the experiment and the theory is defined as follows:

$$f = \left\{ \sum_{i=1}^{N_L} |C_L^{(M)} - C_L(c_{11}, c_{12}, c_{44})| \right\} / 2N_L + \left\{ \sum_{i=1}^{N_R} |C_R^{(M)} - C_R(c_{11}, c_{12}, c_{44})| \right\} / 2N_R, \quad (8)$$

where N_L and N_R are the total numbers of the measured velocities of longitudinal wave and surface wave, respectively. The elastic moduli, c_{11} , c_{12} and c_{44} , are obtained by the direct search method⁽³⁴⁾, that is one of the optimum-seeking method for the minimization of objective function f . The elastic moduli in Table 3 are used as the initial values of the elastic moduli in the calculation. The calculated elastic moduli are indicated in Table 4, on the assumption that the density of specimen in Table 3 is known already. The elastic moduli in Table 4 are in good agreement with that in Table 3. This result shows the validity of the present method.

The problem which we should consider next is the precision of the present method. On the assumption that only one of three elastic moduli is fixed, the function f is evaluated with the others of three elastic moduli changing little by little. The calculated results of the functions f are shown in Fig. 9. The contour lines are drawn on the basis of the minimum value in each function f . It may be considered that the results measured by complex $V(z)$ curve method contain the maximum errors of approximately 8 m/s for longitudinal wave and of approximately 1 m/s for surface waves. In Fig. 9, the intermediate value, 4 m/s, of the maximum error in the experiment is approximately comparable to ± 1 GPa of the calculated elastic

Fig. 9 Results of f for elastic moduli (a) c_{11} versus c_{12} (b) c_{12} versus c_{44} and (c) c_{44} versus c_{11} of gallium arsenide

moduli. This result implies that it is possible for the calculated elastic moduli to be changed, owing to the slight change of the measured velocities for longitudinal wave. On the other hand, when the measured velocities for pseudo Rayleigh wave and Rayleigh wave are used without using the longitudinal wave, the experimental error of the wave velocity of 4 m/s causes the calculation error of approximately ± 3 GPa with respect to elastic moduli of c_{12} .

Consequently, it is understood that the sought results of elastic moduli become stable when the measured velocities for longitudinal wave are used. In order to calculate elastic moduli accurately, it is proposed that the velocities for longitudinal wave should be precisely measured and the velocities of unprecedented acoustic waves for acoustic microscopy should be measured newly.

5. Conclusion

The velocities of acoustic waves for single crystal of cubic system in a local area under 1 mm^2 are measured by the complex $V(z)$ curve method with the line-focus lens. From the measured velocities, the elastic moduli in a local area of the anisotropic material are calculated by the theoretical relation among wave velocities, elastic moduli and density. The results obtained in the present study are as follows:

(1) It is shown theoretically and experimentally that the velocities of acoustic waves could be measured by the complex $V(z)$ curve method with the line-focus lens, in the same way as that by the point-focus lens.

(2) The velocities of the longitudinal wave, pseudo Rayleigh wave and Rayleigh wave on a single crystal of cubic system, could be accurately measured as a function of the azimuthal angle of single crystal. Two velocities of pseudo Rayleigh wave and Rayleigh wave could be simultaneously measured at the particular azimuthal angles. This result suggests the possibility to measure simultaneously the velocities of several kinds of acoustic waves for the anisotropic material that has fewer symmetry axes of the elastic properties. It was found that the signals of the critical angles for the acoustic wave could be recognized clearly when the acoustic impedance of the coupler was selected appropriately compared with the acoustic impedance of the specimen.

(3) The elastic moduli for single crystal of cubic system could be calculated by the optimum-seeking method to fit the theoretical equation related among wave velocities, elastic moduli and density to the measured velocities of longitudinal wave and surface wave.

In the present work, it is shown that the elastic moduli in a local area for single crystal of cubic system, that has many symmetry axes, could be calculated, when the theoretical relation among wave velocities, elastic moduli and density is already given. However, there are many anisotropic materials of which the theoretical relation among wave velocities, elastic moduli and density is unknown or complicated, such as advanced composite materials, functionally gradient materials and so on. In the near future, it is expected to improve the present method or to develop the new method in order to calculate the elastic moduli in these complex problems mentioned above.

Acknowledgment

The authors wish to express their gratitude to Mr. K. Sagara at HOYA Corp. for the supply of glass

specimens.

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