MEASUREMENTS OF THIN-FILM ELASTIC PROPERTIES BY LINE-FOCUS ACOUSTIC MICROSCOPY

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INTRODUCTION

Quantitative acoustic microscopy has been used to measure the velocity of leaky surface acoustic waves (SAWs) [1,2]. This technique measures a V(z) curve, which is a record of the voltage output V of the transducer as a function of the distance z between the acoustic lens and the specimen. Line-focus acoustic microscopy (LFAM) allows the measurement of the SAW velocity in specified directions. In earlier papers, LFAM has been used to determine the elastic constants of isotropic thin films [3,4] and anisotropic thin films [5-7]. The directional variation of the SAW velocity of a thin-layer/anisotropic substrate configuration may be quite different from that of the bare substrate. It follows that this variation can be used to determine the elastic properties of thin films.

The present paper discusses a method to determine not only the elastic constants but also the mass density of an isotropic thin film deposited on an anisotropic solid. The SAW velocities along different directions on the specimen surface have been measured by LFAM. For known film thickness and known material properties of the substrate, the mass density and the elastic constants of the film have been determined from the direction-dependent velocity data. The procedure consists of seeking mass density and elastic constants of the film that yield the best fit in the least square sense of the corresponding calculated SAW velocities to measured SAW velocities. A systematic function-minimization algorithm, the simplex method, has been used to minimize the difference between the calculated and measured SAW velocities.

Amorphous SiC:N films deposited on the (001) plane of a silicon single crystal have been considered in this work. These films were grown under different deposition conditions, hence their material properties are quite different. By using the direction-dependent SAW velocities measured by LFAM, the mass density and the elastic constants of the films have been determined. The effect of the deposition conditions on the elastic properties of the SiC:N films has been investigated.

V(Z) MEASUREMENT

The line-focus acoustic microscope system (Honda AMS-5000) and its operation have been described in detail in Ref.[2]. The acoustic probe consists of a buffer rod with a transducer at one end and a cylindrical lens at the other. The lens is coupled to the specimen through a fluid, usually distilled water. The ultrasonic waves generated by the transducer are focused by the lens into a line-focus beam in the coupling fluid. The focused beam is reflected by the specimen and returned to the transducer to generate a voltage. The voltage output, V, recorded as the specimen is moved vertically towards the lens gives rise to a V(z) curve, where z is the vertical position of specimen. Due to the interference of the specular reflection of the normally incident wave and the radiation of the leaky SAWs

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excited by the wave incident at a critical angle, the V(z) curve shows oscillatory behavior with period Δz. The SAW velocity, v, has a functional relation with this period, which is given as [1,2]

$$v = c_f \left[1 - \left(1 - c_f / 2 f \Delta z \right)^2 \right]^{-1/2} , \quad (1)$$

where c_f is the wave velocity in the coupling fluid and f is the operating frequency. By measuring the V(z) curves for various propagation directions on the surface of the specimen, the directional variation of the SAW velocity can be obtained.

For a cubic-symmetric silicon crystal, SAW velocities obtained from V(z) measurements are shown in Figure 1. The open circles are the SAW velocities on the (001) plane. The solid and dashed lines in Figure 1 are the theoretical velocities of the regular and pseudo-SAWs on a free surface and the dash-dot line gives the value of the anti-plane transverse wave in the bulk solid as described in Ref.[8]. For silicon, the mass density is 2331 kg/m^3 and the elastic constants, c_{11}, c_{12} and c_{44}, are 165.7, 63.9 and 79.6 GPa, respectively [9]. In Figure 1, the angle θ represents the direction relative to the [100] crystalline axis. It is noted that the measured velocities near the 0° and 45° directions are very close to the phase velocities of the regular and pseudo SAWs, respectively. However, near 30° there is a transition range where the measured velocities are neither regular nor pseudo SAWs but a superposition of these waves. This transition range has been discussed in some detail in Ref.[8].

V(Z) MODEL AND SAW VELOCITY

The existence of the transition range causes a difficulty in the use of the measured data to determine the elastic constants, because the directional variation of the measured SAW velocity should be considered as depending not only on the specimen but also on the acoustic probe. This difficulty can be circumvented by comparing the measured velocity with the one calculated from a measurement model for the V(z) curve. Since the V(z) measurement model numerically simulates the measurement procedure, any systemic errors that may occur in the determination of the velocity from experimental V(z) curves will be replicated in the measurement model.

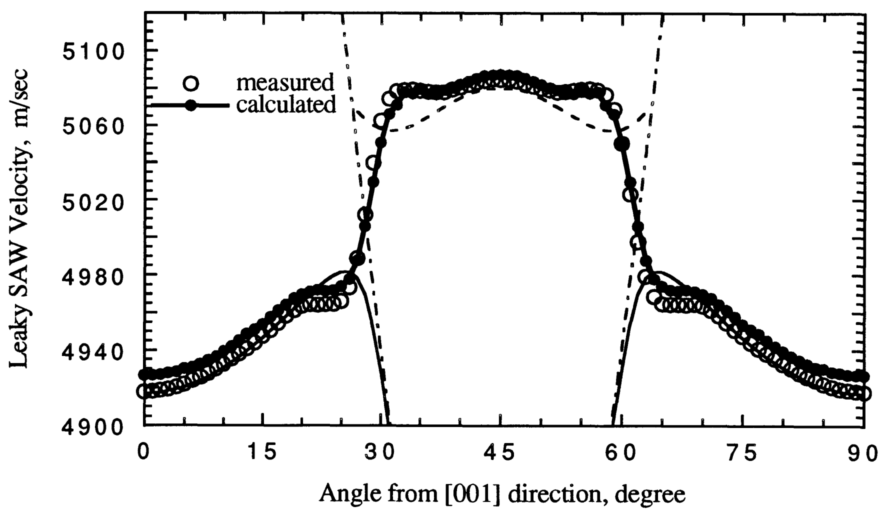

Figure 1 Comparison of the measured and calculated directional variation of the surface acoustic wave velocity on the (001) plane of bare silicon.

A measurement model for the V(z) curve has been described in detail elsewhere [10]. The V(z) curve may be expressed by

$$V(z) = \int_{-\infty}^{+\infty} e^{2iz\sqrt{k_f^2-k_x^2}} L_1(k_x) L_2(k_x) S(k_x) dk_x \quad , \tag{2}$$

where k_f is the wave number in the coupling fluid, $L_1(k_x)$ and $L_2(k_x)$ are characteristic functions of the acoustic lens, and $S(k_x)$ is the reflectance function for the fluid-loaded specimen. In Ref.[10], the functions $L_1(k_x)$ and $L_2(k_x)$ have been described in detail and the $S(k_x)$ for anisotropic solid and anisotropic layer-substrate configurations has been obtained. As a special case from Ref.[10], the $S(k_x)$ for an isotropic-layer/anisotropic substrate configuration has been obtained and used in this work.

To determine the leaky surface wave velocity, the V(z) curves are calculated along different directions. The spacing, Δz, of peaks or valleys in the calculated V(z) curve is determined by a processing procedure analogous to the one used for the experimental V(z) curve, which has been described in Ref.[2]. Upon substitution of the determined Δz in Eq.(1), the SAW velocity is obtained. The solid line with symbols in Figure 1 depicts the SAW velocities for (001) silicon obtained from the calculated V(z) curves. For the determined elastic constants, the corresponding calculated curve follows the measured data very well over the whole range of propagation directions.

SPECIMEN PREPARATION AND MEASUREMENT

The specimens investigated in this work are SiC:N films grown on the (001) plane of silicon crystals. All films have been deposited by the pulsed excimer laser ablation method, as described in Ref.[11]. The films deposited by this method are amorphous, and hence they are isotropic with two elastic constants, Young's modulus, E, and the shear modulus, G. Two deposition parameters are controlled in the film deposition process, the temperature of the substrate and the Nitrogen pressure in the deposition chamber. These deposition parameters for the four thin-film specimens used in this work are listed in Table I. By profilometry the thicknesses of the films have been measured and also listed in Table I. The thicknesses of the films run from 0.68μm to 0.8μm.

For each specimen, the V(z) measurement and the corresponding analysis have been performed for many wave-propagation directions by rotating the specimen at increments of 1^o. Thus, the leaky SAW velocity has been obtained as a function of propagation-direction. The measured velocities are displayed in Fig.2 for specimens #1 to #4. For comparison the measured SAW velocities for bare silicon substrate are also displayed in Fig.2. Since the velocities are symmetrical with respect to the 0^o or [100] direction, and the 45^o or [110] direction, only those between 0^o to 45^o are displayed.

It is noted that in terms of absolute values and shapes the velocity curves for the four thin-film specimens are quite different from each other and from the SAW velocity of the bare silicon substrate. Since the film thicknesses are quite close, the differences in the SAW velocities are caused by the differences in the thin-film material properties. Therefore, inversely, the SAW velocities can be used to determine the material properties of the films.

NUMERICAL INVERSION AND RESULTS

Let $V^m(\theta_i)$ represent the measured SAW velocity along the direction defined by angle θ_i (i = 1,2,.. ,N.) on the surface of the specimen. If the material properties were given, the corresponding velocity, $V^c(\theta_i)$, could be obtained from the calculated V(z) curve as described in Section III. For the thin-film/substrate specimens, the thickness of the film and the material properties of the substrate are already known. Hence, there are three unknowns, namely density ρ, Young's modulus E, and shear modulus G of the film. An inversion procedure is applied to search for a set of values for (ρ, E, G) such that the 'distance' between the measured and calculated velocities is minimized. The 'distance' , D, between $V^m(\theta_i)$ and $V^c(\theta_i)$ is defined as

$$D(\rho, E, G) = \frac{1}{N} \sum_{i=1}^{N} [\, V^m(\theta_i) - V^c(\theta_i\, ; \rho, E, G)\,]^2 . \qquad (3)$$

The inversion procedure consists of seeking a set of (ρ, E, G) that minimizes the function D. A systematic function-minimization algorithm, the simplex method [12], has been used in this work.

The selection of starting values for the simplex method is quite arbitrary. Therefore, the inversion is essentially a process of trial and error. When the starting values are not far away from the minimum, the simplex method quickly converges to the correct values. Different selections of the starting values should yield the same answer. A software routine for the simplex method given in Ref.[13] is used in this paper.

The measured SAW velocities of thin-film specimens #1 to #4, as displayed in Fig.2, have been used for the determination of the mass density and the elastic constants of the films by the inversion procedure. In order to save calculation time, only the data points at increments of 5° from 0° to 45° have been used for the inversion. However, for specimens #1 and #2, the data points between 26° -28° have also been used because the discontinuities of the SAW velocity curves occur around these angles. These discontinuities, which are obviously caused by the presence of the films, are very important features of the films and should be included in the inversion procedure.

The mass densities and the elastic constants of thin-film specimens #1 to #4 determined by the inversion procedure are listed in Table I. As a check, the SAW velocities have been calculated from these determined constants, the known film thickness and the known material properties of the substrate, and are displayed in Fig.2 with solid lines. The calculated curves fit the measured data very well.

From Table I, some useful information about the effect of deposition conditions on the final elastic properties of the film can be obtained. The most significant one is that the

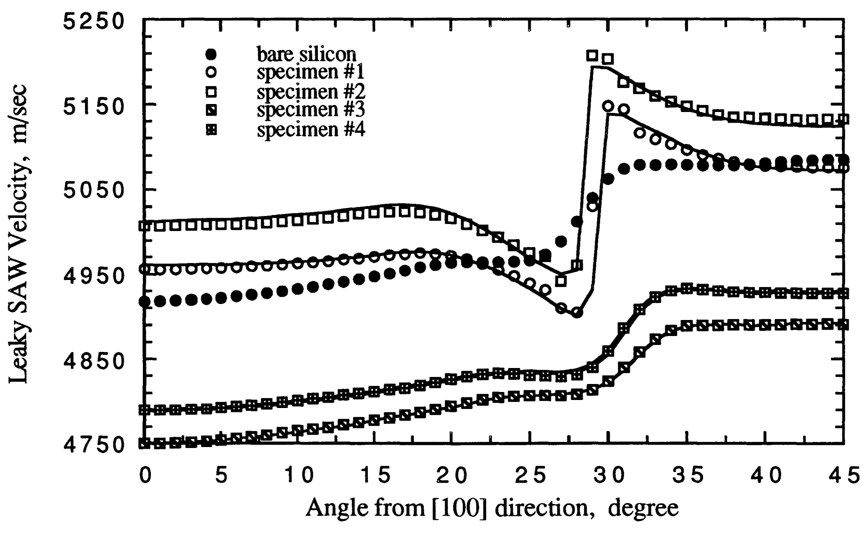

Figure 2 Directional variation of the SAW velocities, measured by LFAM at 225MHz, for SiC:N films deposited on the (001) plane of silicon.

Table I. SiC:N films #1 to #4 deposited on (001) plane of silicon by pulsed laser excimer ablation method.

	Deposition Conditions			Determined Thin Film Properties		
Sample	Temp. °C	N_2 mTorr	Thickness µm	ρ kg/m^3	E GPa	G GPa
#1	24	20	0.675	3590±60	250±10	95±6
#2	650	20	0.65	3130±70	276±10	115±8
#3	24	50	0.80	2250±60	44±2	16±1
#4	24	30	0.68	2570±40	66±2	24±1

elastic properties of the SiC:N films are greatly affected by the N_2 pressure in the deposition process. When the N_2 pressure is increased, the mass density and the elastic constants of the film decrease significantly, as can be seen from specimens #1, #3, and #4. One possible reason is that when the N_2 pressure is increased, more N_2 molecules will be diffused into the film. These N_2 molecules diffused in the film decrease the overall properties of the films. Therefore the higher the N_2 pressure in the deposition process, the lower the mass density and the elastic constants of the film. The temperature of the substrate has less effect on the thin-film properties.

DISPERSION RELATIONS

For a thin-film/substrate configuration the SAW velocity is a function of the frequency. The relation between velocity and frequency, which is represented by a dispersion curve, depends on the material properties of the film as well as the material properties of the substrate. The dispersion curves of the thin-film specimens #1 to #4 have been investigated to check on the material properties of the SiC:N films obtained from the inversion of directional variation of the SAW velocity at 225MHz.

To obtain the dispersion curves for specimens #1 to #4, the operational frequency of the LFAM had to be varied for the V(z) measurements. The LFAM is designed and calibrated at 225 MHz. Changing the frequency induces some measurement errors. To understand the errors, the SAW velocities for a bare (001) silicon substrate have been measured by LFAM at 5 different frequencies, 135, 165, 195, 225, 255 MHz. It is well known that for a bare solid the SAW velocity should be the same at different frequencies. The measured SAW velocities at different frequencies are displayed in Fig.3 for wave

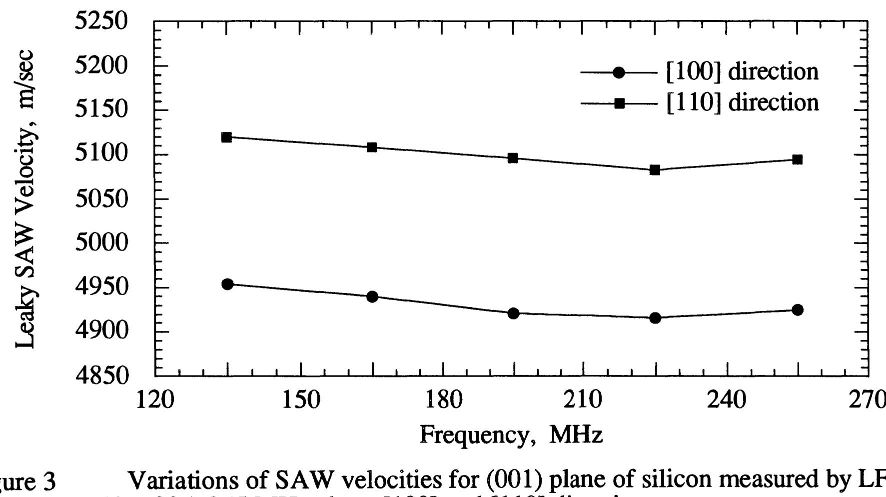

Figure 3 Variations of SAW velocities for (001) plane of silicon measured by LFAM at 135, 165, 195, 225, 255 MHz along [100] and [110] directions.

propagation along the [100] and [110] directions. Because of systemic errors, the velocities at different frequencies show a variation from the velocity measured at 225MHz. The variation is considered as the measurement error of the LFAM at frequencies other than 225MHz.

The dispersion curves of specimen #1 to #4 have been measured by LFAM at 135, 165, 195, 225 and 255 MHz for propagation along [100] and [110] directions. After subtracting the measurement errors, the corrected dispersion curves for specimen #1 to #4 are displayed with symbols in Fig.4(a) and 4(b) for the [100] and [110] directions, respectively. The theoretical dispersion curves of leaky SAW for specimens #1 to #4 based on the mass densities and elastic constants determined in Section V are displayed in

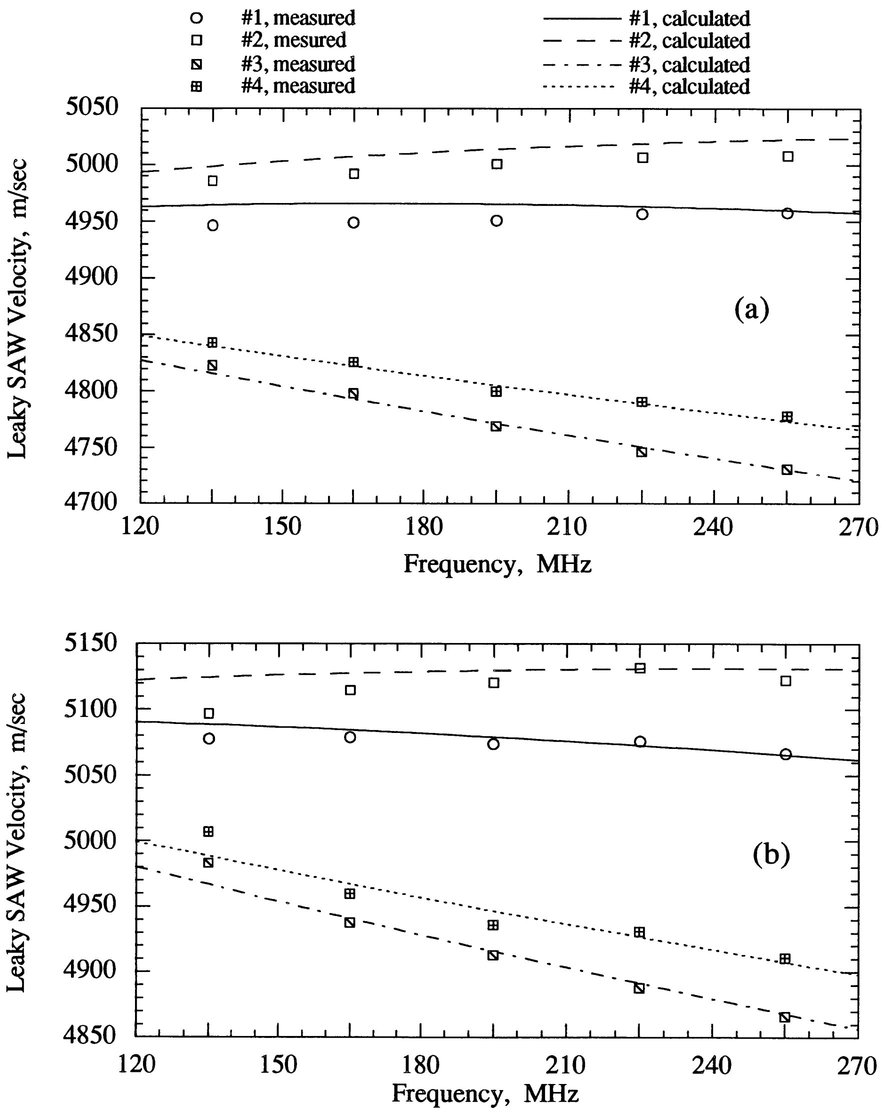

Figure 4 Dispersion curves of SAW velocity for SiC:N films #1 to #4 deposited on (001) plane of silicon, (a) along [100] direction and (b) along [110] direction.

Fig.4(a) and (b) with lines. Good comparisons between the experimental data and the calculated ones can be observed. This agreement provides an additional check on the determined mass densities and elastic constants of the SiC:N films.

SENSITIVITY OF SAW VELOCITIES TO THIN FILM PROPERTIES

It is desirable to check the sensitivity of the SAW velocities to variations of the mass density and the elastic constants in order to understand the limitation of the inversion procedure. Determining the parameter to which the measured data is less sensitive will be more difficult and less accurate.

Figure 5 shows the variation of the SAW velocity for thin-film specimen #1 when one of the material properties of the film, ρ, E or G, is decreased by 10% while the others are kept unchanged. The velocities are determined by the V(z) model described in Section III with the mass density and the elastic constants as determined in Section V. It is noted that the SAW velocity is sensitive to all three parameters ρ, E and G.

CONCLUSION

A non-destructive, in-situ method to determine the mass density and the elastic constants of a thin film deposited on a anisotropic solid has been presented. The directional variation of the SAW velocity of thin-film/substrate configurations has been obtained by line-focus acoustic microscopy. To determine the material properties of the film, the measured SAW velocities have been compared with theoretical ones calculated by a V(z) model. The mass densities and the elastic constants of amorphous SiC:N films deposited by the pulsed laser excimer ablation method have been determined. The correlation between deposition conditions and the properties of the films has been investigated.

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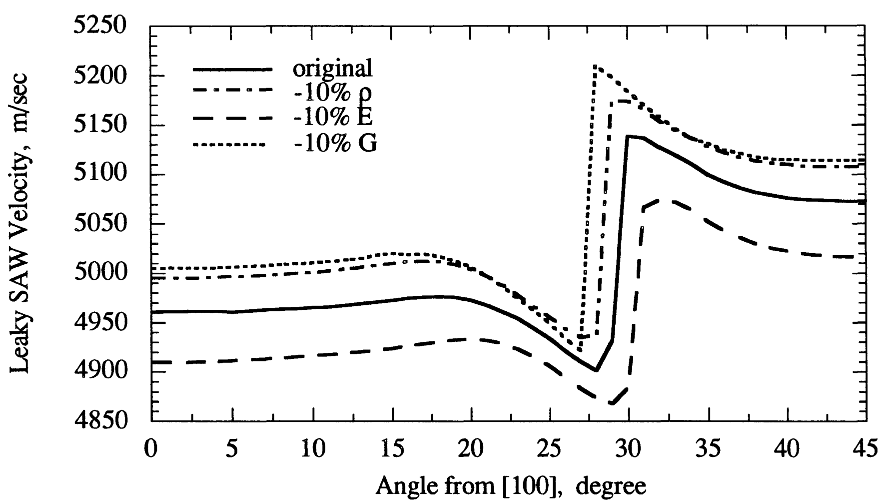

Figure 5 Influence of change of mass density ρ, Young's modulus E and shear modulus G of SiC:N film #1 on the directional variation of the SAW velocity.

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