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Determination of mechanical properties of porous silica low-k films on Si substrates using orientation dependence of surface acoustic wave

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1. Introduction

We have already demonstrated that mechanical properties of thin films are nondestructively characterized by measurement of laser-pulse-generated surface acoustic wave (SAW) and the method has been applied to determination of Young's modulus (E) and Poisson's ratio (v) of porous silica low-k films [1]. However, it is extremely difficult to simultaneously measure both the parameters particularly for thinner films. In this paper, we have improved the measurement accuracy by comparing SAW propagating in [110] and [100] directions of the silicon wafer.

2. Theoretical calculation

It has already been shown by theoretical calculations that dispersion curve and phase velocity of SAW depend on the propagation direction in relation with the crystalline axis of underlying Si substrate [2, 3]. Thus, in order to investigate whether or not the orientation dependence can be used to improve the measurement performance, we calculated phase velocity v_1 of SAW propagating along low-k films on Si substrates, for which parameters such as film thickness, density, Young's modulus and Poisson's ratio are set to be 200nm, 1.0g/cm³, 4GPa and 0.25, typical values of ordinary low-k films. Next, the phase velocity v_2 was calculated for various Young's modulus in the range of 2-6 GPa and Poisson's ratio of 0.05-0.45. The phase velocity differences obtained using the equation,

$$\Delta v = v_1 - v_2(x). \quad x: \text{ various E or } \nu \quad (1)$$

are shown in Figs. 1 and 2, where x stands for various values of E or v.

It is seen in Fig. 1 that the curves for the SAW along the [110] orientation of the Si substrate (a) are quite similar to those along the [100] orientation (b). This indicates that the dependence of dispersion curve on Young's modulus of the film is not so sensitive to the propagation direction. In contrast, differences between the two propagation directions are clear for the dependence on the Poisson's ratio as shown in Fig. 2. These two different behavior allows us to accurately determine values of Young's modulus and Poisson's ratio by simultaneous measurements of SAW propagating along the [110] and [100] orientations of the Si substrate.

3. Experimental

We applied our SAW methods to a porous silica low-k film. Third harmonics of selected 30 ps light pulses from a

mode-locked yttrium-aluminum-garnet (YAG) laser were line-focused onto the specimen to generate SAW. Surface displacement caused by the SAW propagation was detected by a line-shaped piezoelectric transducer at two different distances from the SAW generation line. Density and thickness of the film were determined by x-ray reflectance and spectroscopic ellipsometry analyses. 210.9nm of thickness and 1.042g/cm³ of density are obtained.

4. Results and Discussion

SAW dispersion curves measured along Si [110] and [100] directions for a porous silica low-k film are shown in Fig. 3 (solid lines). Phase velocity is slower in [100] direction than in [110] because the elastic constants of Si have anisotropy. We applied the least square method for determining values of E and v from the two dispersion curves shown in Fig. 3. Figure 4 shows the result of the least square fitting, where there is a clear minimum point. Thus, we obtain $E = 5.3 \pm 0.1$ GPa and $v = 0.259 \pm 0.032$.

A series of porous organic silica films are prepared by changing the ratio of TEOS (tetraethylorthosilicate) and DMDEOS (dimethyldiethoxysilane) in the precursor liquid solution. The films were spin-coated on (100) Si substrates and culminated at 400°C in air ambient. The thickness and density of the resulting films are summarized in Table I. The above double-orientation SAW analysis method was applied to these samples, and the results of mechanical properties are shown in figure 5. The developed analysis method enabled us to detect how the mechanical properties differ depending on the sample preparation conditions; it is clearly seen in this case that both E and v increase with decreasing the amount of DMDEOS.

5. Conclusion

We have succeeded in improving measurement accuracy of mechanical strength of thin films on Si substrates by simultaneously analysis of SAW propagating two different crystalline Si orientations. The method enabled us to sensitively detect the variation of Young's modulus and Poisson's ratio depending on the preparation conditions.

References

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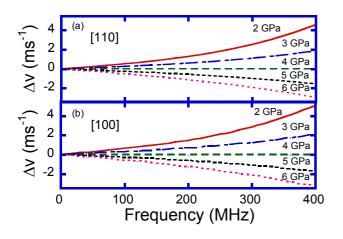


Fig. 1 Phase velocity differences with various values of Young's modulus. In the calculation, the thickness, density and Poisson's ratio of the film are set to be 200nm, $1.0g/cm^3$ and 0.25. Reference parameter of Young's modulus is 4GPa. (a) is for SAW propagating in the [110] orientation of Si substrate, and (b) is for the [100] direction.

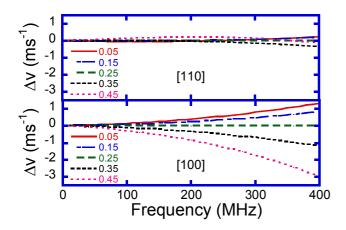


Fig. 2 Phase velocity differences with various values of Poisson's ratio. In the calculation, the thickness, density and Young's modulus of the film are set to be 200nm, $1.0g/cm^3$ and 4GPa. Reference parameter of Poisson's ratio is 0.25. (a) is for SAW propagating in the [110] orientation of Si substrate, and (b) is for the [100] direction.

Table I Density and thickness of the series of porous organic silica films.

DMDEOS Ratio	Density (g/cm ³)	Thickness (nm)
1/4	1.071	296.2
1/5	1.043	210.9
1/6	1.120	227.1
1/8	1.096	294.7
1/16	1.190	270.4

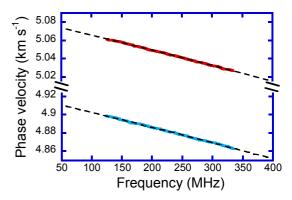


Fig. 3 Measured results of phase velocity of SAW propagating along [110] and [100] directions of Si substrates (solid line) are compared with the fitted theoretical calculations (dashed line) for a porous silica low-k film. The horizontal axis is the frequency of SAW. The fitting was carried out by the least-square determination of Young's modulus and Poisson's ratio of the low-k film.

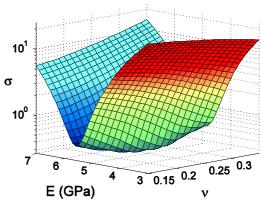


Fig. 4 Fitting behavior of Young's modulus and Poisson's ratio for the data shown in Fig. 3. The vertical axis is the square sum deviation σ between the measured data and the calculated results for the SAW phase velocity with Young's modulus and Poisson's ratio as parameters.

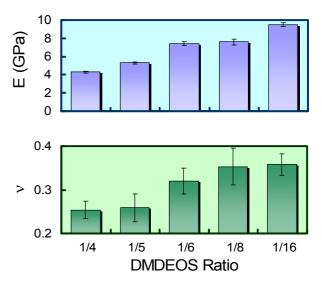


Fig. 5 Measured results of Young's modulus and Poisson's ratio for the series of porous organic silica low-k films shown in Table I.