Determination of the Mechanical Properties of Thin Periodic Porous Silica Films by Laser-Generated Surface Acoustic Wave Technique

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1. Introduction

In order to reach the dielectric constant (k) of the low-kmaterial less than 2.0, it is inevitable to introduce nanopores into dielectrics. Recently, periodic porous silica films have much attention as a candidate of low-k films applied to the copper dual-damascene interconnect technology [1]. The hardness of the thin low-k film is a very important parameter in the copper/low-k process integration. In this work, we developed the laser-generated surface acoustic waves (LSAWs) technique to determine the mechanical properties of several periodic porous silica thin films. Compared to the conventional nanoindentation technique, LSAWs method shows its advantages on nondestructive and accurate technique for hardness determination for fragile ultra-thin films. The LSAWs technique was improved by introducing piezoelectric twin-transducer to ensure the correct position of the surface acoustic wave detector, resulting in the accurate measurement. The periodic porous silica films were produced under different conditions and partly treated with tetraethylorthosilicate (TEOS).

2. Experimental

Periodic porous silica thin films were deposited by spin-coating with 2000-6000rpm on a p-type Si (100) substrate by a sol-gel process from TEOS, cationic surfactant, acid catalyst and solvent (water, alcohol) at room temperature. The films were aged at 453K for 1 hour and then heated up to 673K in a vacuum ($<1.5 \times 10^{-4}$ Pa) or in air. Some of the films were reinforced with additional silica by adsorbing TEOS vapor at 453K, and then heated up to 673K in an air atmosphere. The TEOS treatment of periodic porous silica eliminates the shrinkage of the film, while the film without this treatment shows 10 – 30 % thickness shrinkage [2].

Surface acoustic waves (SAWs) are dispersive on the layered structure. LSAWs technique acquires Young's modulus of the thin film by fitting the dispersive SAW velocity obtained from the experiment with those from theoretical computations. For the structure of the low-*k* film on Si substrate, the frequency dependent SAW velocity (v) is determined by the Young's modulus (E_f), density (ρ_f), Poisson's ratio (σ_f), thickness (h_f) of the low-*k* film and the density (ρ_s), elastic constants (c_{11} , c_{12} , c_{44}) and crystal

structure of the Si substrate. In this connection, LSAW is accurate because the influence from the hard Si subtract is eliminated, which cannot be avoided in the nanoindentation technique. Fig. 1 shows the schematic diagram of LSAWs with a newly designed apparatus piezoelectric twin-transducer detector. In our experiment, third-harmonic light pulses (wavelength: 355nm) of the Nd: YAG laser were employed as the SAW excitation source. The laser beam was focused into a line shape on the sample through a cylindrical lens. Broadband SAWs were generated thermoelastically by the absorption of laser pulse energy at the layer/substrate interface. After propagating for a distance of a few millimeters, SAWs were detected by the piezoelectric detector, which consists of two identical titanium wedge-shaped transducers arrayed parallel in one line. Compared to the general detector used in the ordinary LSAWs technique, this twin-transducer ensures the correct position of the SAWs detector, which is a key factor to influence the measurement accuracy. This is because the experimental conditions for obtaining SAWs dispersion curve shall be consistent with assumptions in the theoretical computation. The SAWs assumed in theoretical calculation are straight-crested in the sense that there are no variations of any of displacements in a direction parallel to the free surface and perpendicular to the direction of propagation [3]. Fig. 2 shows the detected piezoelectric signals of SAWs by transducer 1 and 2 of the detector. The phase shift of two SAW signals implies the position of the detector should be further adjusted until such shift disappears (in the most ideal case), so that the straight-crested surface waves can be detected accurately.

3. Results and Discussions

Fig. 3 shows the Young's modulus determination of several periodic porous silica films by best fitting experimental dispersion curves with those got from theoretical computation. Experimental dispersion curves were obtained by a Fourier transformation process on two SAW signals detected at different wave propagation distances [4]. Theoretical curves were computed by using known film density and thickness determined accurately by the X-ray reflectance and spectroscopic ellipsometry. Table 1 presents determined Young's moduli as well as the known thickness and density of periodic porous silica films. The

Table 1 Young's modulus determination of porous periodic silica films of $2.2 \le k \le 2.5$ and a thermal silica film.

Sample	Thickness (nm)	Density (g/cm ³)	Young's modulus (GPa)
Porous periodic silica I	186	1.15	1.2
Porous periodic silica II	255	0.93	1.0
TEOS-treated Porous periodic silica	338	1.14	1.5
Standard thermal silica	995	2.22	72.2

determined value of 72.2 GPa for the standard thermal SiO_2 film verified the accuracy of our measurements. The results imply that mechanical property of periodic porous silica is improved by the TEOS treatment, and influences of the fabricating process and material treatment could be sensitively detected by the LSAW technique.

Fig. 4 shows an example of the dispersive SAW velocities depending on the Young's modulus of low-*k* film. Once we have a database of such dispersion graphs related to the film thickness, density and Young's modulus, the fitting procedure for the Young's modulus determination is very simple

4. Conclusions

LSAW technique was improved by introducing the newly designed twin-transducer to correctly detect SAWs signals which are consistent with SAWs assumed in the theoretical computation, resulting in the accurate experimental SAW dispersion curve. The Young's moduli of porous periodic silica films were determined by this technique. The TEOS treatment on this low-k film improves its mechanical property. It is found that influences of fabricating process and material treatment on Young's moduli of fragile low-k films could be sensitively detected by this measurement.

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Fig. 1 The schematic diagram of LSAWs apparatus for Young's modulus determination.



Fig. 2 SAW signals detected by twin-transducer for checking the correct position of the detector.



Fig. 3 Young's modulus determination by fitting experimental (solid line) with theoretical curves (dashed line).



Fig. 4 3-dimentinal diagram of SAW velocity depending on the film Young's modulus.