Measuring the Young's Modulus of Ultralow-κ Materials with the Non-Destructive Picosecond Ultrasonic Method

L.L. Chapelon, D. Neira and J. Torres - STMicroelectronics
J. Vitiello - Philips Semiconductors & LPM INSA
J.C. Royer - CEA-Leti
D. Barbier - LPM INSA
F. Naudin, G. Tas, P. Mukundhan and J. Clerico - Rudolph Technologies

ABSTRACT

Young's modulus, which is a measure of elastic strength, provides a good predictor of an interlevel dielectric (ILD) film's ability to withstand chemical mechanical polishing (CMP) and packaging stresses. Picosecond ultrasonics offers a high-throughput, non contact method to measure Young's modulus that is compatible with inline process monitoring. Discussion of this technique and its capability to measure on PECVD non porogen porous MSQ films with good correlation with nanoindentation and ellipsometric porosimetry (EP) measurements will be presented.

INTRODUCTION

Copper and low-κ dielectric materials are required to decrease line resistance and parasitic capacitance, reducing interconnect RC delays and allowing higher device speed and better performance. The shift to copper has proceeded at a rapid rate, but challenges associated with the integration of low-κ materials have slowed their introduction. The main reason is that the mechanical properties of these dielectric films are considerably different from the SiO_2 or fluorinated silicate glass used at the 180 or 120 nm technology nodes.

The International Technology Roadmap for Semiconductors predicts that the dielectric constant (κ-value) of ILD materials will be in the range of 2.7 to 3.0 at the 65 nm technology node, dropping to 2.5 to 2.8 at the 45 nm node, and 2.1 to 2.4 at the 32 nm node[1]. One primary method being used to achieve these lower κ-values is to incorporate porosity into the ILD material. Introducing pores can result in degradation of the material's mechanical properties that may lead to reliability issues.

Young's modulus, which measures a material's elastic strength, is the primary governing property in low-κ film crack initiation and propagation[2,3]. Reducing the modulus of the ILD can result in integration failures such as interfacial delamination and cohesive fracture during CMP and packaging steps. Previous studies have also shown that improvement of the low-κ film's modulus results in better electromigration characteristics of Cu/low-κ structures[4]. Figure 1 shows the dramatic decrease in modulus with the shift to low-κ materials[5]. Therefore, determining the Young's modulus of these materials during development, and maintaining the modulus during production, can be an important predictor of a device's reliability.

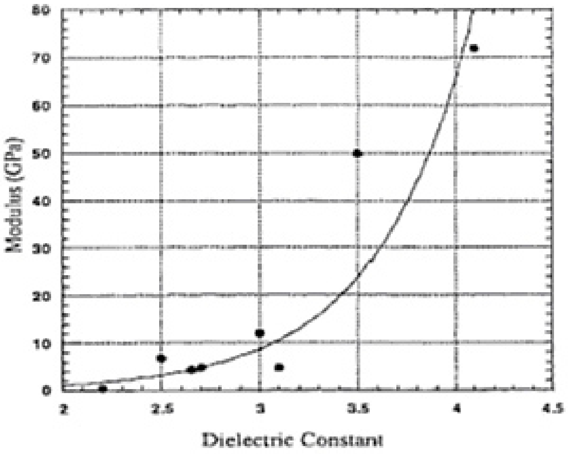

Figure 1 *- Lowering the κ-value of an ILD material improves device performances but results in a lower modulus value and therefore a reduced ability to withstand mechanical stress.*

The traditional techniques for measuring modulus, nanoindentation[5] or bulge/bend tests[6], are not appropriate for inline monitoring as they are relatively slow and destructive. Studies have shown that nanoindentation results may not be reliable when measuring porous low-κ materials for a variety of reasons that include substrate effects, where the nanoindentor is sensitive to the stiffer underlying substrate while nominally measuring the thin, softer, overlying film, tip-film interactions, where the film densifies under the nanoindentor tip, and viscoelasticity[7]. Surface Acoustic Wave (SAW) technology has also demonstrated capability for measuring modulus[8].

Picosecond ultrasonics is currently being used for production monitoring of ILD modulus at the 90 nm technology node. The capability of this technique is being extended to measure the modulus of the ultralow-κ (ULK) films that will be used in future technology nodes. These capabilities were recently tested during the process development stage of ULK films and the results were compared against other techniques.

EXPERIMENTAL

Porous MSQ Samples

As discussed previously, increasing the porosity of the material can reduce its κ-value. To achieve ULK-values, PECVD non porogen porous MSQ films in the κ-value range of 2.0 to 2.65 and with porosity ranging from 0% to 60% were generated[10]. These ULKs may be considered for sub-65 nm technology nodes. The samples ranged in thickness from 2000 Å to 1 µm and were characterized by EP for percent porosity, as well as picosecond ultrasonics and nanoindentation for modulus.

Picosecond Ultrasonic Method

The picosecond ultrasonic technique uses a 0.1 psec laser flash to raise the temperature (typically 5 to 10°C) of a small region (5x7 µm area) on the sample's surface[11]. The pump pulse is transmitted through the transparent ILD to the underlying metal or silicon substrate. The opaque substrate absorbs energy from the pump pulse, launching a longitudinal strain pulse (sound wave) that travels up through the transparent ILD at the speed of sound. The strain causes a local change in the index of refraction of the ILD. The partial reflection of the probe beam from the moving sound wave, combined with the partial reflection from the film surface, leads to destructive and constructive interference at the detector (Fig. 2).

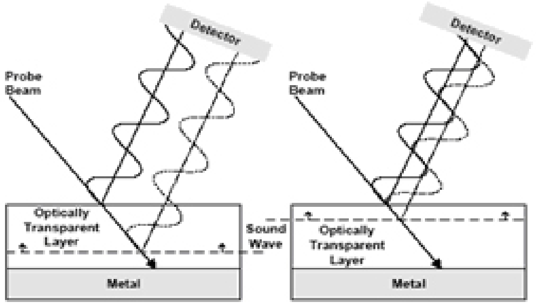

Figure 2 *- The pump pulse passes through the transparent ILD, but is partially absorbed by the underlying opaque layer, creating a sound wave (dashed line) that travels up through the ILD. The sound wave causes a local change in the refractive index that partially reflects the pump beam. This results in destructive (left) and constructive (right) interference at the detector.*

As a result of this time dependent interference, the measured signal oscillates with a period, τ, from which the sound velocity (V) in the material can be determined by:

$$V = \frac{\lambda}{2n\tau\cos\phi} \quad (1)$$

where n is the index of refraction, λ is the wavelength, and Ø is the angle of refraction.

The wavelength of the strain pulse is much smaller than the lateral dimensions of the sample and is assumed to be constrained laterally. By definition, the Young's modulus is a measure of the elastic stiffness (c) of the material when a sample is free to deform laterally[12]. Elastic stiffness is given by:

$$c = \rho V^2 \quad (2)$$

where þ is the density (known value for the low-κ material).

Young's modulus (Y) and elastic stiffness can be related using Poisson's ratio (ν) and is calculated as shown:

$$Y = \frac{(1-2\nu)(1+\nu)^2}{(1-\nu)}\rho V^2 \quad (3)$$

The value for Poisson's ratio is assumed to be constant for the MSQ films in this study.

Fig. 3 is an example of a picosecond ultrasonic measurement signal (black curve) and the best fit model (gray curve) from a dense low-κ SiCOH material used at the 90 nm technology node. The period of the interference oscillations is used to determine the sound velocity in the material as given by equation (1). The index of refraction can be accurately measured with a transparent film metrology system. Using known values for density and Poisson's ratio, the modulus is then calculated using equation (3).

Thickness of the ILD films can also be simultaneously obtained by determining the time it takes for the sound wave to travel through the ILD film. In Fig. 3, the phase change that occurs at around 200 psec results from reflection of the sound at the film surface and can be used to determine the thickness of the low-κ film.

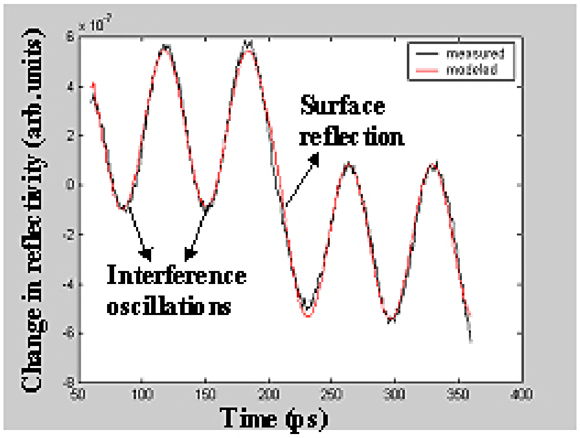

Figure 3 *- The measured signal from a 5000 Å thick SiCOH film. The interference oscillation period depends on the sound velocity in the material. The phase change at approximately 200 psec is caused by the sound wave traveling through the film and reflecting from the top surface. This can be used to determine the thickness of the film.*

RESULTS AND DISCUSSION

Picosecond Ultrasonic Performance

In general, the modulus value should be uniform across the wafer. During development of new low-κ materials, when process conditions are not fully optimized, information about within wafer uniformity is valuable. Picosecond ultrasonic measurements take approximately 2-3 seconds per point, making it possible to rapidly obtain contour maps or measurements from multiple sites across a wafer.

Twenty-five point maps were made to determine the modulus uniformity. Table 1 shows representative data from all 25 measurement sites tested for two porous MSQ films. The standard deviation for within wafer-uniformity for MSQ1 is approximately 5.6% and for MSQ2 is approximately 10%. These types of measurements can be used to help optimize the deposition process to reduce the amount of variation and to insure the desired modulus value is achieved. In addition, once an ILD film is selected for production, if a process excursion occurred that resulted in this type of within wafer or wafer to wafer variation, it could be quickly identified and corrected. Table 2 summarizes average modulus values obtained from 25-point measurements on all sample wafers. The average modulus varied from 1.14 GPa to 4.85 GPa.

	Porous MSQ1 Modulus (GPa)	Porous MSQ2 Modulus (GPa)
1	4.20	2.33
2	3.76	2.87
3	4.42	2.44
4	4.01	2.31
5	3.89	2.21
6	3.97	2.24
7	3.84	2.28
8	3.90	2.34
9	3.95	2.77
10	4.26	2.32
11	3.89	2.79
12	4.53	2.97
13	4.44	2.49
14	4.20	2.94
15	3.92	2.41
16	3.98	2.55
17	4.34	2.56
18	4.24	2.54
19	4.01	2.42
20	3.96	2.41
21	3.98	2.50
22	3.82	2.62
23	3.86	2.91
24	4.25	2.15
25	4.43	2.10
Min	**3.76**	**2.1**
Max	**4.53**	**2.97**
Average	**4.08**	**2.50**
Std Dev	**5.56%**	**10.07%**

Table 1 *- 25-point wafer uniformity data for porous MSQ films. The standard deviation for within wafer-uniformity was approximately 5.6% for MSQ1 and 10% for MSQ2.*

Slot	Thickness (Å)	Avg Modulus (GPa)
7	2000	4.08
8	3000	2.50
9	3000	3.92
10	3000	4.85
11	4400	3.79
12	5600	1.14
13	5600	1.64
14	5600	2.09

***Table 2** - Young's modulus results from various porous MSQ films processed under different conditions. The modulus value was obtained by averaging 25 measurements across each wafer.*

The picosecond ultrasonic results were compared to nanoindentation results as shown in Fig. 4. Modulus values determined with the nanoindentor were consistently higher (approximately three times) than the picosecond ultrasonic measurements. It is likely that the nanoindentor overestimates the modulus of these thin porous films. This is attributed to effects of measuring a soft film on a stiff substrate and the modification to the film surface by the nanoindentor tip that has been observed in other studies[8]. The correlation between the two techniques, however, is excellent.

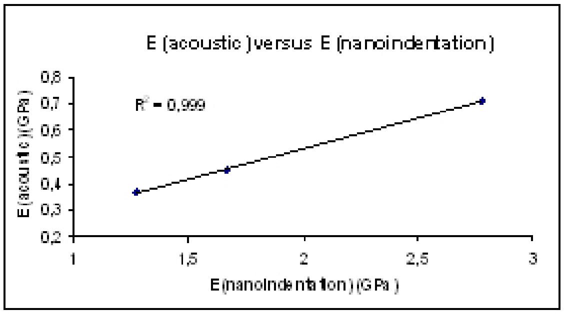

***Figure 4** - The correlation between the experimental picosecond ultrasonic modulus results and those of a nanoindentor are excellent. Note that the nanoindentor results are high compared to the picosecond ultrasonic results, which is likely due to the influence of the stiff substrate.*

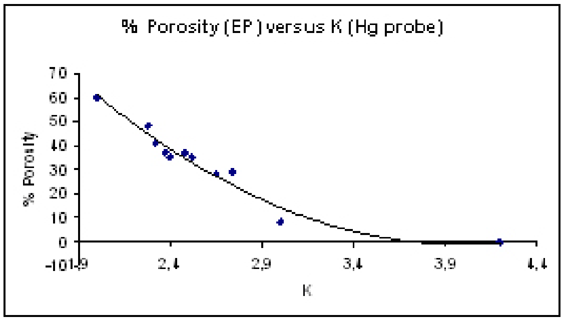

***Figure 5** - Porosity (as measured with Ellipsometric Porosimetry) vs. κ-value (as measured with a mercury probe) of several low-κ generations. Pores were introduced into low-κ to reduce the permittivity in order to improve circuit performance in terms of RC delay for the next technology nodes.*

Porosity and κ-value Comparisons

The previous results demonstrate picosecond ultrasonics capability to measure and map modulus of the porous MSQ films tested. But for high-confidence process control use, the picosecond ultrasonic results need to be consistent with the percent porosity and κ-value results. The relationship between porosity and κ-value for several generations of low-κ films is shown in Fig. 5. The percent porosity was measured with EP and the κ-value with a mercury (Hg) probe. As expected, increasing the percent porosity results in a lower κ-value.

Increasing the porosity is also expected to decrease the mechanical strength. The MSQ films' modulus, as measured with picosecond ultrasonics, was consistent with this assumption. Fig. 6 shows a plot of the measured modulus against EP percent porosity. As the porosity increased from approximately 30 to 60 percent, the modulus decreased from approximately 5 GPa to 1 GPa. This range in modulus values could have a significant impact on the reliability of these films.

While porosity and the κ-value are related (Fig. 5), the critical trade-off that manufacturers must make when selecting an ILD material for future generation devices is between the κ-value and mechanical strength. The picosecond ultrasonics results were therefore compared directly against the κ-value for a wide range of films, as shown in Fig. 7.

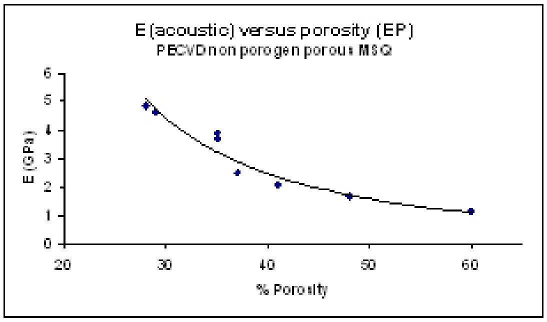

***Figure 6** - Experimental Young's modulus as measured by picosecond ultrasonics of PECVD non porogen porous MSQ dielectrics vs. porosity (as measured with Ellipsometric Porosimetry). As expected, the modulus increases when porosity decreases.*

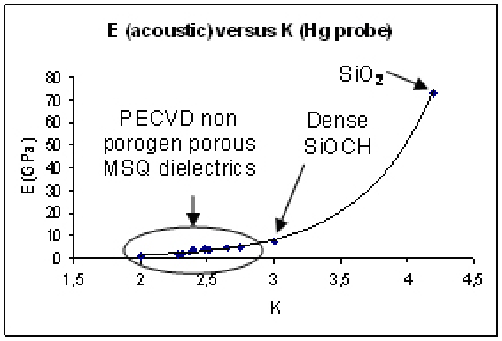

Figure 7 *- Experimental Young's modulus measurements as determined by picosecond ultrasonics of porous and dense dielectrics vs. κ-value as measured by mercury probe. The trend is as expected from the literature as shown in Fig. 1.*

The trend observed is consistent with other studies[5] and compares well with Fig. 1. The modulus values are significantly different for the SiO_2, at approximately 70 GPa, the dense low-κ SiCOH, at approximately 10 GPa, and the porous ULK materials shown in Table 2, at 5 GPa or less. Picosecond ultrasonics evidences the link between porosity, electrical, and mechanical properties of low-κ materials. Porosity decreases κ-value but makes the film softer with a more fragile nature.

CONCLUSION

Picosecond ultrasonics provides a high-throughput, non destructive technique to monitor modulus values during process development and is compatible with inline processing. This technique has shown its ability to measure on porous MSQ materials in the κ-value range of 2.0 to 2.65 that may be adopted at the 65 nm technology node and below and show good agreement with percent porosity, κ-value and nanoindentation.

REFERENCES

[1] International Technology Roadmap for Semiconductors: 2005 Edition, Semiconductor Industry Association, 2005.

[2] G.Xu, J.He, E.Andideh, J.Bielenfeld and T. Scherban, "Cohesive strength characterization of brittle low-κ films," Proceedings of the IEEE 2002 IITC, p57-59, 2002.

[3] R.F. Cook and E.G. Liniger, "Stress-corrosion cracking of low-dielectric-constant spin-on-glass thin films," J.Electrochem.Soc, Vol. 146 No. 12, 4439-4448 (1999).

[4] K.Lee, E.T.Ogawa, S.Yoon, X.Lu and P.S.Ho, "Electromigration reliability of dual-damascene Cu/ porous methylsilsesquioxane low k interconnects," Appl. Phys. Letters, Vol. 82, No. 13, March 2003, p 2032-2034.

[5] T. M. Moore, et. al., AIP Conf. Proc. 550 (2001) 431.

[5] G.M. Pharr and W.C. Oliver, "Measurement f thin film mechanical properties using nano- indentation," MRS Bulletin, pp.28-33, July 1992

[6] Xiang, X. Chen, and J.J. Vlassak ,"The mechanical properties of electroplated Cu thin films measured by means of the bulge test technique", Mat. Res. Soc. Symp. Proc., Vol. 695, pp.L4.9.1-6 2002

[7] C. M. Flannery and D. C. Hurley, "Laser ultrasound: an inspection tool of soft porous low-dielectric constant films for microelectronic interconnect", Review of Nondestructive valuation, Vol. 22, 2000.

[8] M. Gostein, A. Mazurenko, and A. A. Maznev, "Measuring Young's modulus of low-κ dielectrics using surface acoustic waves", MICRO, June, 2004.

[10] L.L. Chapelon, V. Arnal, M. Broekaart, L.G. Gosset, J. Vitiello, J. Torres, Microelectronic Engineering 76 (MAM 2004) 1-7.

[11] C.Thomsen, H.T. Grahn, H.J. Maris, and J. Tauc, "Surface generation and detection of phonons by picosecond light pulse," Physical Review B, Vol. 34, no. 6, pp.4129-4138, 1986.

[12] W.P. Mason, Physical Acoustics and the Properties of Solids, D.Van Nostrand Co. Inc., New York, 1958