Young's Modulus of Dielectric 'Low-k' Materials

Application Note

Jennifer Hay

Introduction

In digital circuits, insulating dielectrics separate the conducting parts (wire interconnects and transistors) from one another. As components have scaled and transistors have gotten closer and closer together, the insulating dielectrics have thinned to the point where charge build up and crosstalk adversely affect the performance of the device. It is this reduction in scale which drives the need for insulating materials with lower dielectric constant. A 'low-k' material is one with a small value for dielectric constant relative to silicon dioxide (SiO_2)—a former dielectric of choice. The dielectric constant of SiO_2 is 3.9. This number is the ratio of the permittivity of SiO_2 divided by permittivity of vacuum, $\epsilon_{SiO_2}/\epsilon_0$, where $\epsilon_0 = 8.854 \times 10^{-6}$ pFμm. There are many materials with lower dielectric constants, but few of them can be suitably integrated into a semiconductor manufacturing process [1].

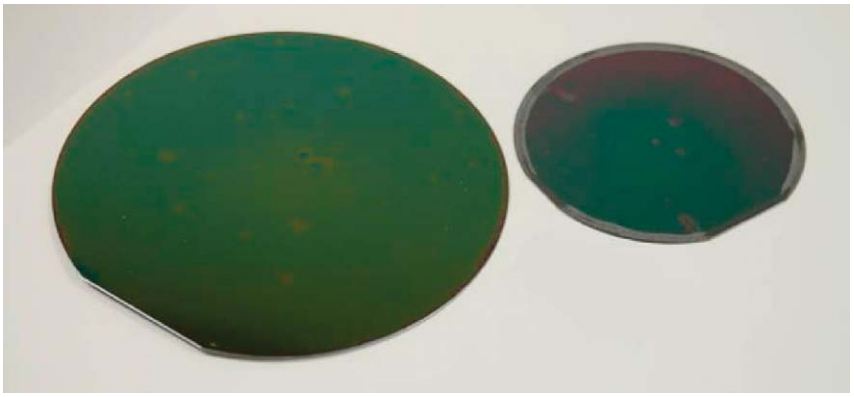

Figure 1. Whole silicon wafers, coated with low-k materials.

At the extreme, dry air (20C, 1 atm) has a dielectric constant of 1.00059 [2], but dry air cannot keep conducting materials mechanically separated, so it cannot be used as an insulator. But as one incorporates material for structure, the dielectric constant also increases. So the optimization problem in materials development for semiconductors is to lower the permittivity of the dielectric material as far as possible without compromising mechanical integrity, as quantified by the Young's modulus. Generally, processes purposed for reducing permittivity (such as pore introduction) also have the effect of reducing Young's modulus.

For over ten years now, we have provided value to the semiconductor industry by giving them a way to measure Young's modulus of low-k materials as deposited on silicon wafers as shown in Figure 1. This is the single largest industrial application for our technology. This note reports the results for two dielectric materials supplied by a loyal customer.

Theory

Instrumented indentation testing (IIT) is a technique for measuring the mechanical properties of materials. It is a development of traditional hardness tests such as Brinell, Rockwell, Vickers, and Knoop. Instrumented indentation testing is similar to traditional hardness testing in that a hard indenter, usually diamond, is pressed into contact with the test material. However, traditional hardness testing yields only one measure of deformation at one applied force, whereas during an IIT test, force and penetration are measured for the entire time that the indenter is in contact with the material. Nearly all of the advantages of IIT derive from this continuous measurement of force and displacement. Instrumented indentation testing is particularly well suited for testing small volumes of material such as thin films, particles, or other small features. It is most commonly used to measure Young's modulus (E) and hardness (H) [3, 4]. The Young's modulus for a material is the relationship between stress and strain when deformation is elastic.

Using data from a single instrumented indentation test, the reduced modulus (E_r) is calculated as

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad \text{(Eq. 1)}$$

where A is the contact area and S is the elastic stiffness of the contact. The elastic stiffness of the contact may be determined in two different ways. It may be determined semi-statically as the change in force with respect to displacement when the indenter is first withdrawn from the sample, because this part of the test manifests purely elastic recovery. It may also be determined dynamically by oscillating the indenter [4, 5]. If S is determined by the first (semi-static) method, then modulus can only be realized at the maximum penetration. But if S is determined by the second (dynamic) method, then modulus can be determined as a continuous function of penetration depth. This ability to determine properties as a continuous function of penetration depth is particularly useful when testing dielectric films on silicon [6]. Once the elastic stiffness S has been determined by either method, Young's modulus is calculated from the reduced modulus as

$$E = \left(1-\nu^2\right)\left[\frac{1}{E_r} - \frac{1-\nu_i^2}{E_i}\right]^{-1}, \quad \text{(Eq. 2)}$$

Where v_i and E_i are the Poisson's ratio and Young's modulus of diamond, respectively, and v is the Poisson's ratio for the test material. Although calculation of Young's modulus (Eq. 2) requires knowing the Poisson's ratio of the sample (v), the sensitivity is weak. Sensitivity analysis reveals that a generous uncertainty of 40% in the Poisson's ratio manifests as only a 5% uncertainty in the Young's modulus. Typically, a value of 0.18 is used for dielectrics.

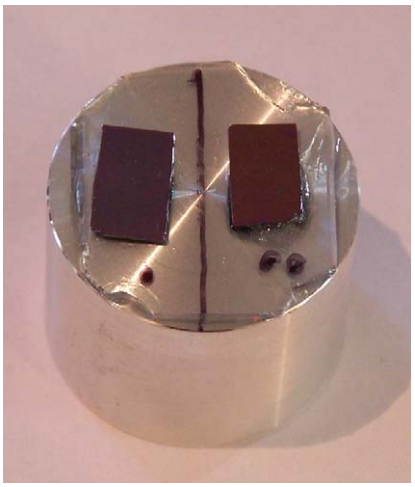

Figure 2. Two low-k samples, as mounted for testing in the G200.

Experimental Method

The Agilent G200 Nanoindenter was used to test two low-k films on silicon; the thickness of the first film was 1007 nm and the thickness of the second film was 445 nm. Figure 2 shows the two samples mounted for testing. The G300 Nanoindenter could be used to perform the same measurements with the added benefit of using vacuum to anchor the whole wafer for testing. Both platforms (the G200 and the G300) may be used with either of two actuating/sensing mechanisms or "heads": the XP head or the DCM II head. The DCM II head offers the best precision in fundamental measurements of force, displacement, and phase shift. Thus, it is the best choice for testing low-k films. A DCM II head fitted with a Berkovich diamond indenter was used for all tests reported in this note.

Agilent Nanoindenters have been the industry choice for low-k testing precisely because of the *continuous stiffness measurement* (CSM) option, which measures elastic contact stiffness (S) dynamically. With the CSM option, every indentation test returns a complete depth profile of Young's modulus. Using this option, eight tests were performed on each sample. Loading was controlled such that the loading rate divided by the load (P'/P) remained constant at 0.05/sec; loading was terminated when the indenter reached a penetration depth of 200 nm. The excitation frequency was 75 Hz, and the excitation amplitude was controlled such that the displacement amplitude remained constant at 1 nm.

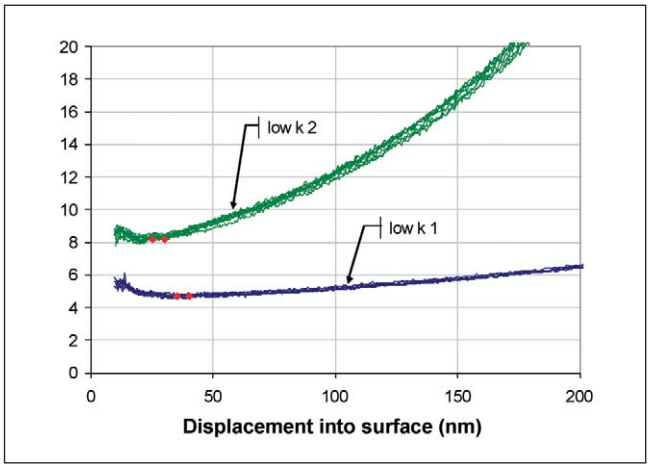

Figure 3. Young's modulus as a function of surface penetration for both samples; all eight tests on each sample are shown. Modulus increases with depth due to increasing substrate influence. Displacement range for citing modulus is bounded by red diamonds.

Sample	Film thickness, nm	Displacement range for cited properties, nm	E, GPa	σ(E), GPa
Low-k 1	1007	35-40	4.69	0.07
Low-k 2	445	25-30	8.23	0.13

Table 1. Summary of results.

Results and Discussion

Figure 3 shows the depth profiles of Young's modulus; the results of all 16 tests (eight on each sample) are shown. The repeatability is remarkable: it is due to the ultra-high precision of the DCM II and the smoothness and uniformity of the samples. Young's modulus increases as a function of displacement, because the silicon substrate increasingly affects the measurement. Because "low-k 2" is a thinner film, the Young's modulus increases more quickly with depth, relative to "low-k 1". Having such profiles, one can easily discern the critical depth at which the substrate begins to affect the measurement, and thus report film properties over a depth range which is shallower than this critical depth.

The following analysis procedure was applied to each sample to report Young's modulus *for the film alone*. First, a representative displacement range was identified. The minimum value of this range should be large enough to be unaffected by surface anomalies. The maximum value of this range should be small enough to be unaffected by substrate. These identified ranges are shown on the graph of Figure 3. For the first test, all readings within this displacement range were averaged to get E_1. Values for subsequent tests were calculated in the same way to get $E_2, E_3, \ldots, E_8$.

Finally, the average and standard deviation were calculated for all $n = 8$ tests as

$$\overline{E} = \frac{1}{n}\sum_{j=1}^{n} E_j \text{, and} \qquad \text{(Eq. 3)}$$

$$\sigma(\overline{E}) = \sqrt{\frac{1}{n}\sum_{j=1}^{n}(E_j - \overline{E})^2} \text{.} \qquad \text{(Eq. 4)}$$

These calculations are fully automated within the regular CSM test method of the controlling software, NanoSuite. The Young's moduli calculated in this way for the two materials are reported in Table 1.

Conclusions

The Young's moduli of "low-k 1" and "low-k 2" were 4.67±0.07 GPa and 8.23±0.13 GPa, respectively. Because it has a higher modulus, "low-k 2" should withstand the rigors of semiconductor manufacturing better.

The Agilent G200 Nanoindenter with a DCM II head is the industry choice for these measurements because of its high-precision, speed, ease of use, and the CSM option, which delivers properties as a continuous function of penetration depth. With the CSM option, the influence of the substrate is easily discerned, thus allowing properties to be cited at depths that are shallow enough to be free of substrate influence. Without the CSM option, Young's modulus can only be realized at the maximum penetration depth. Therefore, the user must know the zone of substrate independence *a priori* in order to wisely specify the maximum indentation depth. Often, such information is not available prior to testing—CSM is valuable precisely because it eliminates the need for such information. The zone of substrate independence can be identified *after* testing is complete.

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