Young's Modulus and Hardness of Thin Low-κ Films Using Nanoindentation

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Abstract

In this work, we used nanoindentation to measure the mechanical properties of two nominally similar low-κ films on silicon, having film thicknesses of 196 nm and 478 nm. The iNano system automatically accounted for the influence of the silicon substrate, so as to measure the properties of the film alone. For the 196 nm film, we measured E = 6.48±0.27; H = 1.22±0.06 (N=10). For the 478 nm film, we measured E = 6.14±0.14; H = 1.17±0.03 (N = 10). Comparing the two films, the differences in E and H were slight, but statistically significant ($p < 0.05$). The greater strength of the thinner film was due to the microstructure caused by the smaller volume constraint.

Introduction

Nanoindentation allows one to measure Young's modulus and hardness of thin films and other small volumes of material. By far, the most common application for nanoindentation is the mechanical characterization of low-dielectric-constant (low-κ) films for microelectronics. In microelectronic chips, conducting lines are separated both electrically and mechanically by low-κ materials. For optimal electrical separation, the dielectric constant, κ, should be as low as possible. However, processes which tend to lower the dielectric constant, such as increasing porosity in the film, also tend to lower the strength of the material. Thus, an important optimization in microelectronic design is to lower the dielectric constant as much as possible while maintaining sufficient mechanical strength. Nanoindentation is the primary means for evaluating the mechanical strength of low-κ materials.

Figure 1. Measuring the mechanical strength of microelectronic materials is the most common application for nanoindentation.

For the purpose of nanoindentation, low-κ films are usually deposited on silicon wafers. When testing such thin films, the fundamental nanoindentation measurements can be substantially affected by the supporting silicon substrate. Consequently, the Young's modulus measured according to the Oliver-Pharr method alone[1] can be artificially inflated, even when the indentation depth is only a small fraction of the film thickness. The iNano indentation system includes an automatic correction for substrate influence, so that the measured Young's modulus is the true value for the low-κ film, independent of the substrate upon which it is deposited[2]. The true Young's modulus of the film, E_f, is calculated as a function of the uncorrected or "apparent" Young's modulus (E_a), the indentation depth (h), the thickness of the film (t_f), the substrate Young's modulus (E_s), and the substrate Poisson's ratio (ν_s):

$$E_f = f\left(E_a, h, t_f, E_s, \nu_s\right) \tag{1}$$

All the parameters on the right-hand side are typically well known for low-κ films.

All iNano systems use a small superimposed oscillation during loading to measure both Young's modulus and hardness as a continuous function of indenter penetration. (Without this superimposed oscillation, one can only measure Young's modulus and hardness at the maximum penetration depth[1].) Being able to see Young's modulus and hardness as a continuous function of penetration depth is tremendously useful when testing thin films, because it reveals the degree to which the substrate is affecting the measured properties, both before and after the correction for substrate influence. When the substrate influence is properly corrected, the measured properties should be substantially independent of indentation depth.

Experimental Method

We tested two samples of low-κ materials provided by SBA Materials, Inc., a leading supplier of advanced electronic materials (*www.sbamaterials.com*). The iNano was used to measure the Young's modulus and hardness of these materials. For these measurements, the system was fitted with a Berkovich indenter, which is the most common indenter used for nanoindentation.

Sample ID	Film thickness (nm)	Refractive Index, n§	Dielectric constant, κ†
A	196.27	1.285	2.215
F	477.51	1.283	2.169

Table 1. Summary of low-κ samples; physical properties measured by SBA Materials.
§Measured at a wavelength of 633 nm.
†Measured at 1 MHz

Ten indentation tests were performed on each sample at room temperature using the test method, "Dynamic CSR for Thin Films". This test method performed the following automated steps at each test site:

1. The indenter face was brought into contact with the film using an approach speed of 30 nm/sec.
2. The indenter was pressed into the surface of the film using a strain rate of 0.2/sec to 50% of the film thickness while simultaneously oscillating with an amplitude of 1 nm and a frequency of 100 Hz.
3. At the peak depth, the force was held constant for a brief dwell period of 1 second.
4. The contact force was reduced over 10 seconds to 10% of the peak force.
5. The force on the indenter was held constant for 80 seconds while the displacement of the indenter was monitored. Since the force was constant, the ideal displacement rate during this segment should be zero. The true displacement rate, which is not identically zero due to thermal drift, was measured during this constant-force segment of the test.
6. The indenter was withdrawn completely, and the sample was moved into position for the next test.

The Hay-Crawford model for extracting film properties requires knowledge of the elastic properties of the substrate, in addition to film thickness. Table 2 summarizes all the analytic inputs used to calculate the mechanical properties of each film.

Input	Value
Drift Correction?	1
Film Thickness	196 or 478
Poisson's Ratio of Substrate	0.2
Young's Modulus of Substrate	170
Poisson's Ratio of Film	0.3
Fraction of Film to Start Avg	0.29
Fraction of Film to End Avg	0.31
Thin Film (1) or Bulk (0)	1

Table 2. Summary of analytic inputs. (Note: Any of these values may be changed post-test, and the results quickly re-calculated.)

Results and Discussion

The superimposed oscillation applied during loading (1 nm, 100 Hz) yields a continuous measure of contact stiffness as the indenter is pressed into the material. In Figure 2, the faded traces (pink and light-blue) show the Young's modulus achieved by applying the Oliver-Pharr method to dynamic stiffness measurement. The x-axis is the indentation depth, normalized by the film thickness.

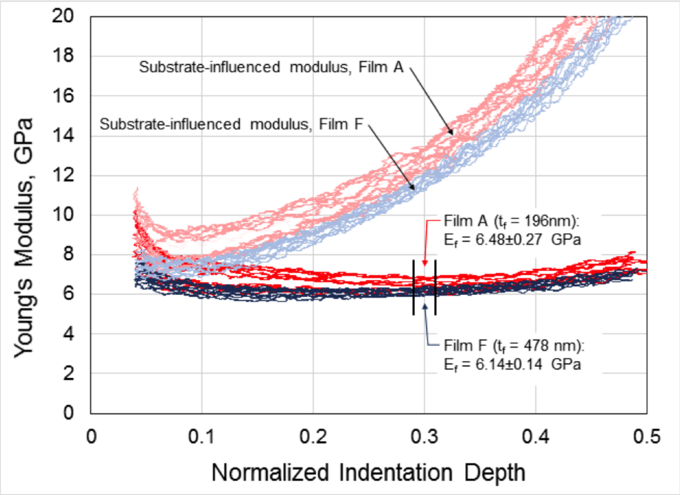

Figure 2. Young's modulus as a function of indentation depth, normalized by film thickness. Each pink trace represents a single test on Film A, and each light-blue trace represents a single test on Film F. The red and blue traces represent these same physical measurements, respectively, only corrected for substrate influence. Using the corrected traces, film modulus is reported around 30% of the film thickness.

When the indentation depth is a small fraction of the film thickness, the Young's modulus is relatively low, but as the indentation depth increases, the measured Young's modulus also increases, due to the increasing influence of the very stiff silicon substrate (E = 170 GPa). Although these values are indeed substrate affected, they are a valuable first-step, because they show the influence for which we must account.

The red and blue traces in Figure 2 demonstrate that the Hay-Crawford model does indeed accurately compensate for substrate influence, because the modulus values are substantially constant as a function of normalized indentation depth. The red and blue traces are the same physical measurements as the original pink and light-blue traces, but they have been further analyzed to account for substrate influence. To report a single value for Young's modulus from each indentation, all the data from each corrected trace are averaged between the limits of 29% and 31%. These limits (which are set by the user as in Table 2) are shown as the solid black lines in Figure 2. Finally, the mean and standard deviation of these values are calculated across all 10 tests to get E_f = 6.48±0.27 for film A and E_f = 6.14±0.14 for film F.

In the field of nanoindentation, it is common practice to assume that indentations which are less than 10% of the film thickness are substantially independent of substrate influences. However, for low-κ materials, such an assumption causes significant error, because the substrate is so much stiffer than the film. Indeed, the values of moduli which are calculated from the faded traces in Figure 2 at 10% of the film thickness are E_a = 8.49 GPa for film A, and E_a = 7.43 GPa. The value for sample A is artificially inflated by 30% and the value for sample B is artificially inflated by 20%! Business decisions based on these inflated values could be devastating for a manufacturer.

The Hay-Crawford model also increases accuracy and repeatability by allowing measurements to be made at larger displacements than would otherwise be used if substrate influence were a concern. Put simply, the user is much more likely to get accurate, repeatable measurements at 50nm than at 20nm. As the indentation depth decreases, the effect of any experimental difficulty is magnified. Such difficulties include surface roughness, tip imperfections, contact detection, and environmental vibrations. The Hay-Crawford model allows substrate-independent measurements to be made using indentation depths as large as 40% of the film thickness.

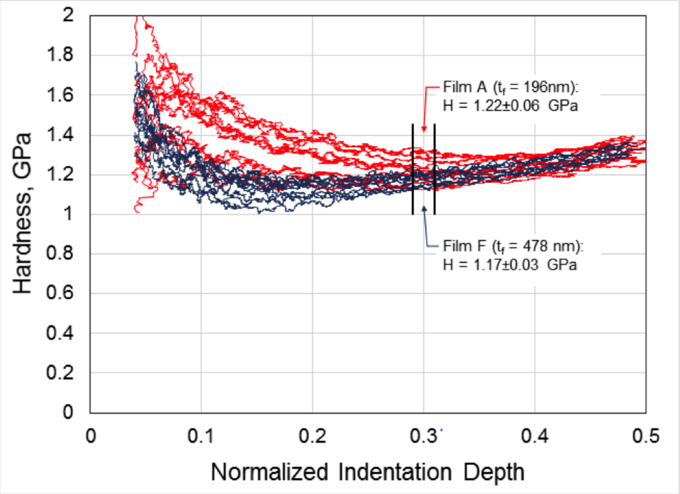

Figure 3. Hardness as a function of indentation depth, normalized by film thickness. Each red trace represents a single test on Film A, and each navy trace represents a single test on Film F. Hardness is relatively insensitive to substrate influence, so no corrective model is needed. Using the original traces, hardness is reported around 30% of the film thickness.

Hardness is much less sensitive to substrate influence, because the zone of plastic deformation is much smaller than the zone of elastic deformation. Figure 3 shows the hardness measured by the Oliver-Pharr method alone. Even though the film is much softer than the silicon substrate, the hardness traces barely increase with normalized indentation depth. Therefore, no correction is needed to account for substrate influence on hardness. To report a single value for hardness from each indentation, all the data from each trace are averaged between the limits of 29% and 31% (between the solid black lines).

Finally, the mean and standard deviation of these individual values are calculated across all 10 tests to get H = 1.22±0.06 for film A, and H = 1.17±0.03 for film F. (Interestingly, at 40% of the film thickness, the traces to seem to converge to nearly identical values.)

Table 3 summarizes all the indentation results for both samples. The differences in E and H between the two samples are small, but statistically significant ($p < 0.05$). The thinner film A has a slighter greater Young's modulus and hardness. Although the films were designed to be identical, the smaller volume constraint for sample A affects the microstructure, and this effect is manifest in the mechanical properties. This conclusion is supported by the slight difference in dielectric constant measured for the two materials. The thicker sample F has a slightly lower dielectric constant, so it is not surprising that it also has a slightly lower modulus, because processes that tend to lower the dielectric constant also tend to lower the modulus.

Sample ID	N	Depth range (nm)	E_f (GPa)	H (GPa)
A	10	56.8 - 60.8	6.48±0.27	1.22±0.06
F	10	138.6 - 148.2	6.14±0.14	1.17±0.03

Table 3. Summary of film mechanical properties measured with the iNano

Conclusions

Nanoindentation is a valuable measurement tool precisely because volume affects microstructure, which in turn affects mechanical properties. The mechanical properties of materials often depend on the volume in which they are forced to exist. The iNano indentation system employs state-of-the-art hardware, electronics, procedures and analyses to give accurate and repeatable properties of very thin films. In this work, we compared two nominally similar low-κ films, and found that the thinner film had a slightly greater Young's modulus. This finding might easily have been skewed or obscured by other commercial indentation systems, especially those which do not employ thin-film modeling, or dynamic indentation during loading.

Acknowledgements

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References

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2. Hay, J.L. and Crawford, B., “Measuring Substrate-Independent Modulus of Thin Films, Journal of Materials Research 26(6):727-738 (2011).