Introduction

Mechanical characterization of low-k films deposited on silicon is the single largest application for instrumented indentation. Thus, Keysight has continually sought test procedures and analyses which improve accuracy and minimize testing time for these kinds of samples.
Typical low-κ wafers are shown in Figure 1. For a long time, the Continuous Stiffness Measurement (CSM) technique was the preferred way to test low-κ films on silicon, because it gave elastic modulus and hardness as a continuous function of indenter penetration\(^1\). Generally, the measured elastic modulus and hardness increased with indenter penetration due to the increasing influence of the rigid silicon wafer beneath the film. In 2011, Keysight incorporated an analytic model which, when applied to CSM data, actually compensated for substrate influence \(^2\)–\(^4\). This improvement allowed accurate and substrate-independent characterization of films as thin as 200nm. In 2012, Keysight introduced a new technique called Express Test which dramatically increased testing speed \(^5\), \(^6\). When combined with the Keysight thin-film model, Express Test also gave substrate-independent results. This application note demonstrates the cumulative benefit of all these improvements for our customers in the semiconductor industry.

Theory

CSM and Express Test are very different kinds of experimental procedures, but if the material is not significantly strain-rate sensitive, the two procedures give the same results for hardness and elastic modulus.

In a CSM test, the indenter is slowly pushed into the material while a small oscillation (~1nm amplitude) is used to sense the instantaneous elastic stiffness of the contact. Elastic modulus and hardness are calculated from the measured quantities of force, indenter displacement, and contact stiffness (derived from the characteristics of the oscillation). Thus, indentations performed with CSM give elastic modulus and hardness as a continuous function of indenter displacement \(^7\). Each CSM indent takes about 5 minutes.

With Express Test, the indenter is rapidly pushed into the surface to a prescribed force or displacement and then withdrawn. The elastic stiffness of the contact is determined from the elastic recovery of the material which occurs as the indenter is withdrawn from the material. Elastic modulus and hardness are calculated from the measured quantities of force, displacement, and contact stiffness (derived from the elastic recovery during unload). Thus, indentations performed with Express Test only give elastic modulus and hardness at the prescribed peak force or displacement. However, each indentation only takes about one second \(^5\).

A practical difference between CSM and Express Test is that with CSM, the user can decide which data is most appropriate for reporting after the test is complete. This is especially relevant for thin-film testing. For example, after testing, the user may decide to report modulus for each indentation by averaging the values in the modulus channel that are associated with penetration depths between 100nm and 200nm. Or, the user may decide that it is better to report modulus by averaging values in the modulus channel which are associated with penetration depths between 250nm and 300nm. The user has the freedom to make such decisions after testing is complete, based on what the continuous measure of modulus reveals about surface abnormalities and substrate influence. With Express Test, however, the user must decide before testing what force or displacement should be used, because each indentation yields properties for elastic modulus and hardness only at the prescribed force or displacement. For example, for a film which is 1000nm thick, the user...

<table>
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<tr>
<th>Sample</th>
<th>tf</th>
<th>NanoSuite Method</th>
<th>Depth limit</th>
<th>No. of indents</th>
</tr>
</thead>
<tbody>
<tr>
<td>low-k 1</td>
<td>1007</td>
<td>G-Series DCM CSM Hardness, Modulus for Thin Films</td>
<td>150</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Express Test for Thin Films</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>low-k 2</td>
<td>445</td>
<td>G-Series DCM CSM Hardness, Modulus for Thin Films</td>
<td>75</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Express Test for Thin Films</td>
<td>44</td>
<td>100</td>
</tr>
<tr>
<td>fused silica</td>
<td>N/A</td>
<td>G-Series DCM CSM Standard Hardness, Modulus, and Tip Cal</td>
<td>500</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Express Test Tip Calibration</td>
<td>varied</td>
<td>400</td>
</tr>
</tbody>
</table>

Table 1. Summary of testing.
might prescribe indentations to 100nm (~10% of the film thickness).

CSM and Express Test are kinds of testing procedures. The Keysight thin-film model is an analysis that can be applied to either CSM or Express Test data. Basically, it accounts for substrate influence on modulus using the known parameters of indentation depth, film thickness, and substrate modulus [2–4]. (Substrate influence on hardness is usually negligible.) In this work, we demonstrate the use of four combinations of procedure and analysis: 1) CSM alone, 2) CSM with thin-film model, 3) Express Test alone, and 4) Express Test with thin-film model. We do this in order to show that the fourth combination (Express Test with thin-film model) is the best choice for quickly and accurately characterizing low-k films on silicon.

**Experimental Method**

**Samples**

Two low-k films on silicon were tested, hereafter referred to as “low-k 1” and “low-k 2”. Low-k 1 had a film thickness of 1007nm and low-k 2 had a film thickness of 445nm. Figure 2 shows the two samples mounted for testing. Fused silica was tested as a reference material.
Equipment and Procedure

The two low-k samples were tested, along with fused silica, in a Keysight lab with a Keysight G200 Nanoin-denter, configured with both the CSM option and the Express Test option. All tests were performed using a DCM II head fitted with a Berkovich indenter. Table 1 summarizes the details of testing. For Express Tests, the peak displacement was prescribed to be 10% of the film thickness, or 100nm for low-k 1 and 44nm for low-k 2.

Results

Table 2 summarizes the most important results of this work, but these results cannot be understood without a careful explanation of Figures 3 and 4.

Figure 3(a) displays modulus results for low-k 1. In this plot, there are four sets of results: Blue lines, green lines, blue symbols and green symbols. The blue lines are the results of the 12 CSM indentations on this sample, where the data have not been corrected for substrate influence. From about 60nm onward, the blue lines gradually increase due to the increasing influence of the silicon substrate. The green lines are the results of CSM testing where the data have been corrected for substrate influence. Because the substrate influence has been accounted and removed, the green lines manifest a constant value with increasing displacement. It is important to understand that the blue lines and the green lines both have exactly the same underlying physical

<table>
<thead>
<tr>
<th>Sample</th>
<th>tf (nm)</th>
<th>Procedure</th>
<th>No. of indents</th>
<th>Testing Time (minutes)</th>
<th>Modulus (1 std. dev.) (GPa)</th>
<th>Hardness (1 std. dev.) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>low-k 1</td>
<td>1007</td>
<td>CSM</td>
<td>12/12</td>
<td>72</td>
<td>4.44 (0.04)</td>
<td>0.687 (0.011)</td>
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<tr>
<td></td>
<td></td>
<td>Express Test</td>
<td>98/100</td>
<td>7</td>
<td>4.44 (0.08)</td>
<td>0.698 (0.015)</td>
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<tr>
<td>low-k 2</td>
<td>445</td>
<td>CSM</td>
<td>12/12</td>
<td>72</td>
<td>7.75 (0.09)</td>
<td>1.150 (0.024)</td>
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<td></td>
<td></td>
<td>Express Test</td>
<td>93/100</td>
<td>7</td>
<td>8.19 (0.18)</td>
<td>1.203 (0.033)</td>
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</table>

Table 2. Summary of results. Both CSM and Express Test values for modulus have been corrected using the Agilent thin-film model.
data. They do not come from different CSM tests; rather, they come from the same 12 indentations. The green lines are simply the result of applying the thin-film model to the data represented by the blue lines. In Table 2, the CSM modulus for low-k 1 (4.44GPa) is the average of all the green lines between 95.7nm and 105.8nm (9.5% – 10.5% of the film thickness).

In Figure 3(a), the blue symbols represent the 100 Express Test indents on low-k 1, where the data have not been corrected for substrate influence. Like their CSM counterparts, the values gradually increase with displacement due to increasing substrate influence. The green symbols represent the same tests where the data have been corrected for substrate influence. Because the green symbols represent substrate-accounted results, they are constant with depth. Again, the green and blue symbols do not represent different indentations, but rather, different interpretations of the same indentations. The green symbols are substrate-accounted and the blue symbols are not. In Table 2, the Express Test modulus for low-k 1 (4.44GPa) is the average of all the green symbols.

Figure 3(b) gives the same information for low-k 2 that Figure 3(a) gives for low-k 1. The lines and symbols should be understood in exactly the same way. In Table 2, the CSM modulus for low-k 2 (7.75GPa) is the average of all the green lines between 42.3nm and 46.7nm (9.5% – 10.5% of the film thickness).
thickness). The Express Test modulus for low-k 2 (8.19GPa) is the average of all the green symbols.

Figure 4 gives hardness results for both low-k samples. Since the measurement of hardness is insensitive to substrate influence, there is no need for a corrective model. Thus, Figure 4 only compares CSM results (blue lines) with Express Test results (blue symbols). In Table 2, CSM hardness is the average of all blue lines between 9.5% and 10.5% of the film thickness. Express Test hardness is the average of all the blue symbols.

Figure 5 gives hardness and modulus for the fused silica reference material. As expected the CSM and Express Test results agree well for this material.

Discussion

Only substrate-corrected values for modulus are reported in Table 2. For the measurement of modulus, the advantages of the thin-film model are clear, whether it is applied to CSM or Express Test data. The model accurately accounts for the influence of the substrate (as evidenced by the fact that modulus is constant with increasing displacement), and because substrate influence is properly accounted and removed, the precise displacement at which measurements are made is less important. In other words, the substrate-accounted modulus is substantially the same, whether evaluated at 10% of the film thickness or 15% of the film thickness.

The agreement between CSM and Express Test is outstanding, considering the profound difference between these two procedures. For low-k 1, there was no significant difference between the properties measured by each method. For low-k 2, there was a slight, but statistically significant difference between the CSM and Express Test properties. Express Test returned slightly higher values for both elastic modulus and hardness. There are several possible explanations for the difference. First, the properties may be slightly different from location to location. The Express Test indentations were performed at a different site than the CSM indentations. No particular effort was made to make the CSM indents near the Express Test indents. More extensive testing would be required to confirm or deny this hypothesis. Another possible explanation is that the difference in test procedure is the cause of the observed difference in properties. If low-k 2 is significantly strain-rate sensitive, then Express Test may simply yield higher values for modulus and hardness due to its faster rate of deformation. However, the most likely explanation is that the CSM results are more susceptible to thermal drift than Express Test results. Thermal drift is the expansion and contraction that occurs over time with temperature fluctuation. Because Express Test is so fast (1 indent per second), the influence of thermal drift is nil. But CSM tests take much longer; the indenter is in contact with the material for about 2 minutes. Even though CSM methods include means for minimizing and accounting for thermal drift, this may still be the best explanation for the slight difference between the CSM and Express Test results for low-k 2.

The measurements on fused silica help us to know that system calibration is not the reason that the Express Test properties are slightly higher than the CSM properties for low-k 2. Around 40nm, the Express Test modulus is exactly the same as the CSM modulus. The Express Test hardness is actually slightly lower than the CSM hardness.
Given the close agreement between CSM and Express Test results, the dramatic speed of Express Test warrants its use over CSM. Express Test is two orders of magnitude faster than CSM. It took about 70 minutes to perform 12 CSM tests, but it took only 7 minutes to perform 100 Express Tests. With Express Test, mechanical characterization may finally be able to keep pace with the demands of the semiconductor industry.

Conclusions

Though both CSM and Express Test offer accurate measurements, Express Test is preferred for testing low-k films on silicon due to its sheer speed. The elastic modulus and hardness of a low-k film can now be determined accurately and robustly in just 7 minutes. With Express Test and a proprietary thin-film model, the Keysight G200 NanoIndenteter offers the most powerful tool in the world for the mechanical characterization of low-k films on silicon.

References

Nano Mechanical Systems
from Keysight Technologies

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