Scratch Testing of Low-k Dielectric Films and a Correlation Study of the Results

Application Note

Introduction

The processing that is required to lower the dielectric constant in a low-k film has the adverse effect of degrading the mechanical properties of the film. Low-k films are subjected to many processes that test the strength of these films and their adhesion to the substrate such as chemical and mechanical polishing (CMP) and wire bonding. It is important for these materials to resist plastic deformation during these processes and remain intact without blistering up from the substrate. Ideally, a dielectric material will have a high hardness and elastic modulus because, traditionally, these two parameters help to define how the material will react when subjected to manufacturing processes. In this application note, scratch tests are preformed on several low-k samples using a ramp-load scratch test. The results from scratch testing and nanoindentation are examined through correlation analysis to better understand the interconnectedness of scratch results.

Samples

Ten low-k samples were provided for scratch testing by SBA Materials, Inc. The films were deposited on silicon substrates by spin coating and their thicknesses varied between samples from 375 nm to 743 nm. Samples were supplied with the elastic modulus and hardness measurements of the films. Mechanical properties data were collected using an Agilent Nano Indenter G200 with a special test method for measuring the substrate independent properties of low-k dielectric films. This test method is described elsewhere [1]. A summary of the sample thicknesses and mechanical properties is given in Table 1.

Sample	Film Thickness nm	Elastic Modulus Gpa	Hardness Gpa
Sample A	500.7	6.61	1.08
Sample B	617	8.1	1.25
Sample C	743	8.05	1.28
Sample D	470	8.25	1.25
Sample E	526	8.66	1.26
Sample F	641.6	6.9	1.11
Sample G	488.9	7.13	1.14
Sample H	375	8.66	1.26
Sample I	423	***	***
Sample J	650	8.15	1.28

Table 1. Film thicknesses and mechanical properties of the low-k film samples.

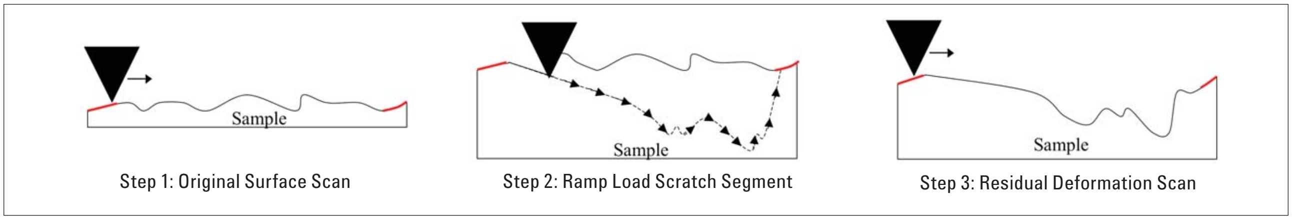

Figure 1. Diagram of the three-step ramp-load scratch test. Red lines show the areas of pre and post profile scans used to perform leveling of the 3 steps.

Test Methodology

All of the scratch tests were performed on the Nano Indenter G200. The Nano Indenter G200 is powered by electromagnetic actuation to achieve unparalleled dynamic range in force and displacement. The instrument's unique design avoids lateral displacement artifacts during the scratch process. Using the G200, researchers can measure Young's modulus and hardness in compliance with ISO 14577, in addition to scratch and wear properties. Deformation can be measured over six orders of magnitude (from nanometers to millimeters).

A ramp-load scratch test was used to conduct three tests on each wafer in three locations — this totaled nine tests for each sample. In a ramp-load scratch test, a tip is brought into contact with the sample; then, the tip is loaded at a constant loading rate while simultaneously translating the sample. Prior to and following the scratch test, a single-line-scan of the surface topography is completed for comparing the original surface to the deformation caused by the scratch test. Therefore, each scratch test consists of three steps: a single-line pre-scan of the area to be scratched, the ramp-load scratch test, and a final scan to evaluate the residual deformation. Before and after each step, a pre-scan and a post-scan, usually equal to 20% of the scratch length, is performed so that the software can automatically align the data in the three steps. The original and residual single-line scans allow for the evaluation of deformation mechanisms and the quantification of deformation. The scratch process is diagramed in Figure 1.

When performing scratch testing on any sample set, it is critical that all test parameters and tip geometries remain consistent throughout the samples being compared. This ensures that qualitative comparisons can be made using the resulting data. The test parameters used in testing the low-k materials are listed in Table 2.

The tip chosen for conducting the scratch tests was a cube-corner tip with a tip radius that was, nominally, less than 20 nm. A cube-corner tip creates a triangular projected contact with the sample; this tip geometry creates high levels of stress in the material during the scratch. Scratches can be performed either face-forward or edge-forward when using a pyramid shaped indenter.

Scratch Length	300 µm
Scratch Velocity	30 µm/s
Maximum Scratch Load	3 mN
Scratch Direction	Face-forward

Table 2. Parameters used in the ramp-load scratch test.

Scratching face-forward with the cube-corner tip acts like a snow plow and pushes the material out of the way, while edge-forward cuts the material like a knife. A diagram of a cube-corner tip is shown in Figure 2. The low-k samples were tested using the cube-corner tip positioned so that it scratched face-forward.

Results and Discussion

Film Failure

All of the low-k films failed in a similar manner; these films exhibited plastic deformation up to a critical point where blistering of the film occurred. Following blistering, complete failure of the film occurred and the substrate was scratched for the remainder of the test. For complete understanding of the mode of failure exhibited during the scratch tests on the low-k samples, a typical scratch test was analyzed using scanning probe microscopy, which was available on the Nano Indenter G200 by using the Nano Vision option.

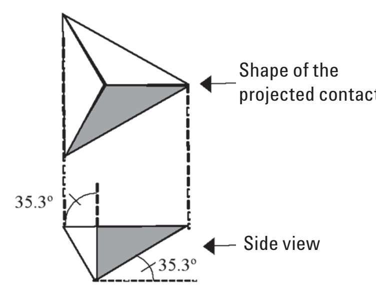

Figure 2. Diagram of a cube-corner tip.

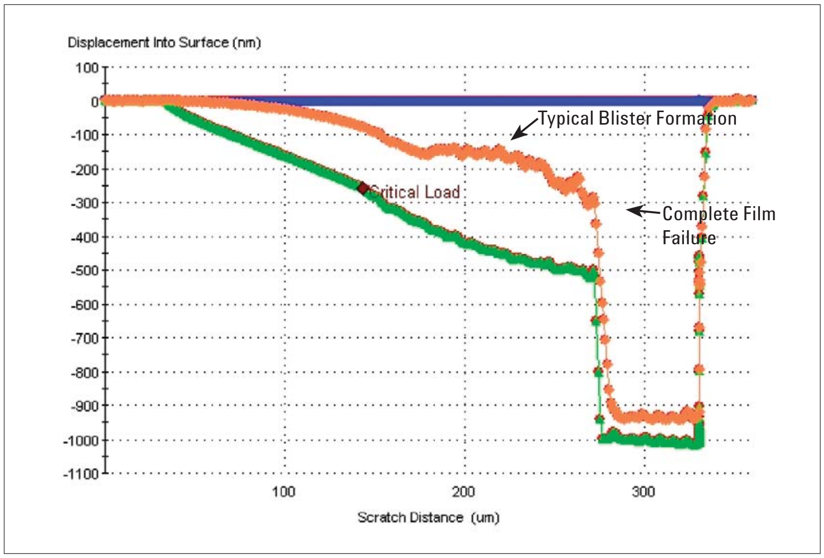

Figure 3. The *Displacement into Surface* versus *Scratch Distance* data for the scratch that was used for imaging to further examine failure; the blue trace is the *Original Surface Topography*, the green trace is the *Scratch Curve*, and the orange trace is the *Residual Deformation*. The locations of blistering and total film failure are labeled.

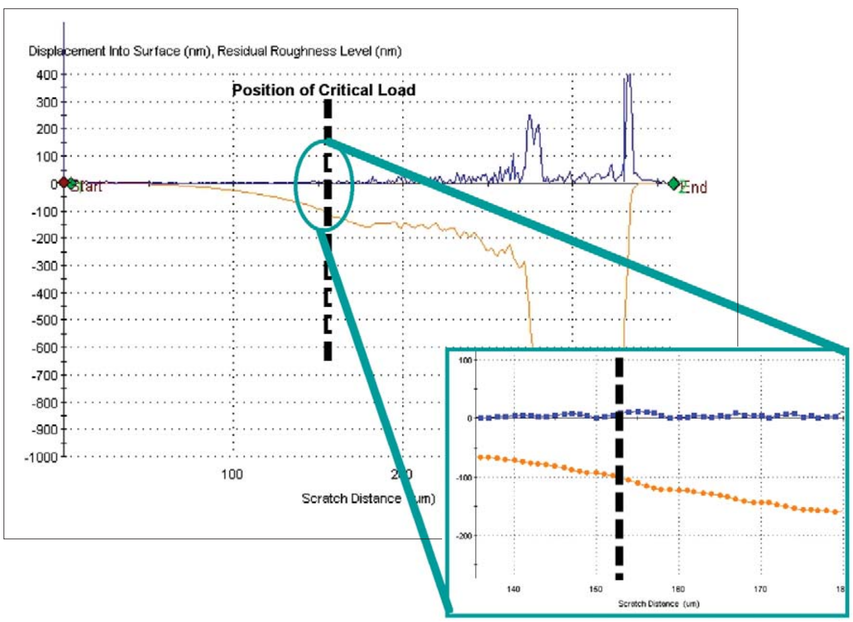

Figure 4. *Residual Roughness* (blue) and the *Residual Deformation* scan (orange) versus *Scratch Distance*.

The Nano Vision option allows imaging through the use of a high precision peizo translation stage; lateral resolutions and flatness of travel is better then 2 nm. This system allows quantitative imaging and high precision targeting for the investigation of material properties.

The scratch test that was used for the imaging analysis took the film to failure and the displacement results are shown in Figure 3. The *Critical Load* was chosen automatically by the software through examination of the residual displacement curve. In the calculation of the critical load point, the Agilent NanoSuite software examines the rate of change of the displacement in the residual scan (this is the orange trace in Figure 3) and, using these data, extrapolates the displacement curve for the next data point based on the change in the scratch distance. The actual observed displacement is compared to the predicted displacement and the magnitude of the differential is recorded as the *Residual Roughness Level*. Figure 4 shows a plot of the *Residual Roughness* along with the *Residual Deformation* of the scratch; for presentation, the *Residual Roughness* channel has been multiplied by three so that it will appear on a graph with the displacement curves. The *Critical Load* was placed in the location where the *Residual Roughness* was greater than 3 nm – hence, this is the location where the anticipated displacement is in error as compared to the actual displacement by greater than 3 nm. The tolerance for placing the *Critical Load* is determined by the roughness detected during the surface scan of the pre-profile.

To examine the blistering and failure of the low-k films, the scratch test performed in Figure 3 was imaged with

Nano Vision. Figure 5 shows the top and side views of the scratch test that was performed in Figure 3. Here it is clear that a large amount of material has blistered just prior to failure of the film. The scale shows that the film blistered up approximately 400 nm above the surface of the film. Typically, blistering is not measured well by the residual scan of a scratch test because the film supports very little load parallel to the scratch direction and is usually pushed down by the profiler. However, Nano Vision was used to scan the scratch deformation in the transverse direction which allowed the detailed observation of the blister. To ensure that blistering was being observed as opposed to pile-up, another scan at the start of blistering was conducted. Figure 6 shows the scan of the scratch test at the start of blistering. It is evident from the scan that the residual scratch depth increases until blistering occurs; then, the residual deformation in the film is lifted upward off of the substrate due to blistering. If pile-up had occurred, the residual scratch depth would have continued to increase as the scratch test progressed.

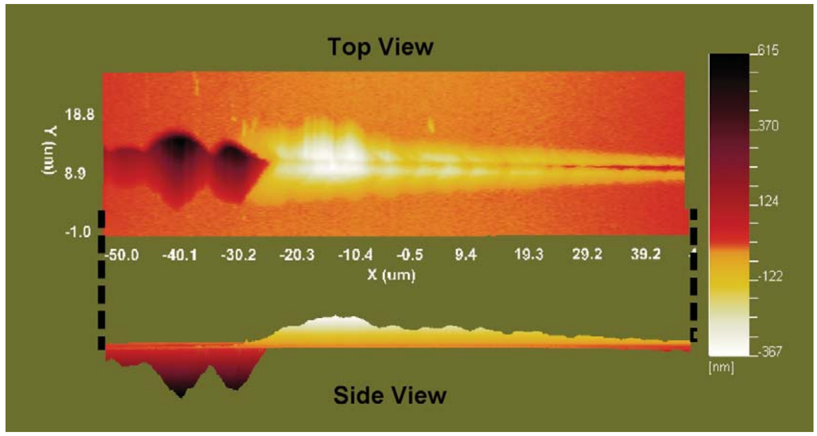

Figure 5. Typical blistering and failure in the low-k films. Imaging of the scratch was performed using the Nano Vision option on the Nano Indenter G200. Notice that the film has blistered up approximately 400 nm from the surface.

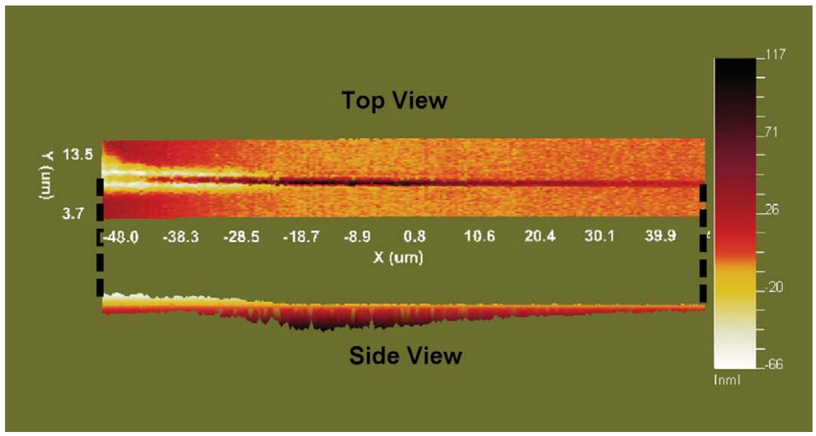

Figure 6. Typical scratch tests in the low-k films showing the start of blistering. Notice that the residual scratch depth increases until blistering occurs. Following the start of blistering, the residual deformation in the film has been lifted upwards.

Sample	Critical Load Value mN	Depth at Critical Load nm	Total Deformation µm2	Percent Penetration at Critical Load %	Elastic Deformation up to Critical Load %
Sample A	0.97±0.029	197±17	10.6±1.0	59.5±5.1	74.5±0.7
Sample B	1.306±0.083	261±23	18.9±2.0	73.9±6.5	75.4±3.3
Sample C	1.22±0.022	257±9	17.4±0.8	73.6±2.6	77.4±2.2
Sample D	1.045±0.055	204±16	11.4±1.1	51.2±4.0	66.3±3.6
Sample E	1.157±0.052	233±12	15.1±1.3	75.9±3.9	72.8±2.1
Sample F	1.067±0.019	225±6	13.1±0.6	55.8±1.5	78.5±1.2
Sample G	1.025±0.063	212±21	11.6±1.4	54.0±5.3	74.7±1.9
Sample H	0.811±0.063	162±8	7.3±0.4	44.2±2.2	72.1±4
Sample I	0.927±0.048	168±5	7.4±0.6	45.4±1.4	72.4±1.5
Sample J	1.345±0.041	278±12	20.5±1.2	76.1±3.3	74.3±3.2

Table 3. Summary of scratch results.

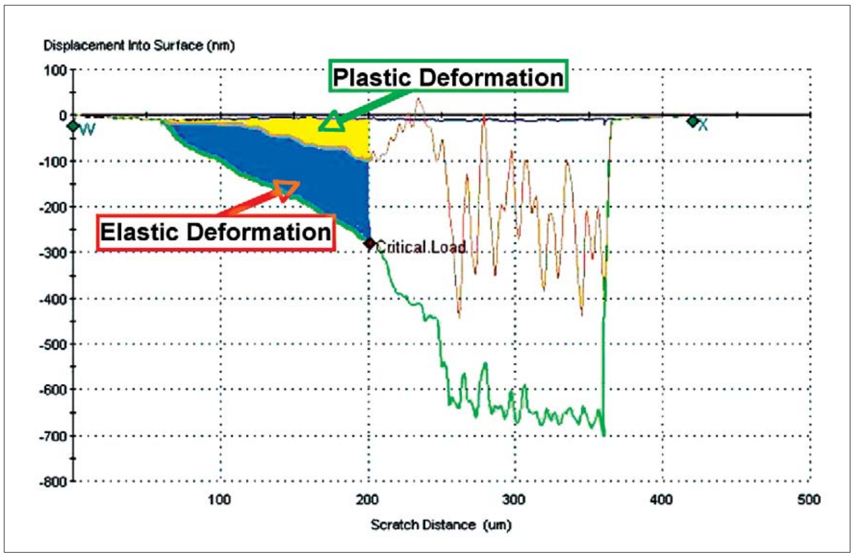

Figure 7. Elastic and plastic deformation of a scratch test performed on Sample J; the *Elastic Deformation* is shaded in blue and the *Plastic Deformation* is shaded in yellow. The film thickness for Sample J is 650 nm.

Scratch Results

A summary of the scratch results for all ten samples is provided in Table 3. The sixth column of this table provides the amount of elastic deformation that occurred during the scratch up to the point of film failure. Elastic and plastic deformation during the scratch test is determined by measuring the areas between the *Scratch Curve* and the *Original Surface Topography* and between the *Residual Deformation Scan* and the *Original Surface Topography*. This new parameter, *Percent Elastic Deformation up to Critical Load*, provides a measure of the films resistance to permanent deformation and provides a more complete evaluation of the films performance by quantifying not only the load at which film failure occurs, but also the type of deformation occurring up to film failure. The areas of elastic and plastic deformation are shaded on the scratch curves of Figure 7.

Figures 8 and 9 graphically display the results of *Critical Load* and *Total Deformation*, and the *Percent Penetration/Elastic Deformation* and *Elastic Modulus* for the four top performing samples based on the results of *Critical Load* — the four top performing samples were B, C, E, and J. Here it is shown that there are subtle differences between the top performers. Samples B and J, which had the highest critical loads of all the samples, show no significant statistical difference in the result for *Critical Load*, but Sample J possessed a much smaller standard deviation in its results; thereby suggesting that Sample J is more repeatable and predictable.

Sample C possessed the second lowest *Total Deformation* and the highest amount of *Elastic Deformation* of these top performing samples; this is quite significant since this sample was the thickest sample tested by approximately 100 nm. So, even though Sample C had the most material available for accommodating deformation, it showed the highest resilience of all the samples. In addition, its performance in *Critical Load* was on par with the other samples. Similarly, the results for Sample E are significant because the film thickness for this sample is thinner then any of the other top performers by approximately 100 nm. While Sample E did not have the highest *Critical Load*, it did possess the least amount of *Total Deformation* and had one of the highest results for *Percent Penetration at Critical Load*.

After presentation of the results to the manufacturer, it was disclosed that samples E and J had the same chemical composition but used different solvents for dilution during the spin coat process. Therefore, either the difference in film thickness or the use of different solvents caused the two samples to have different *Critical Load*

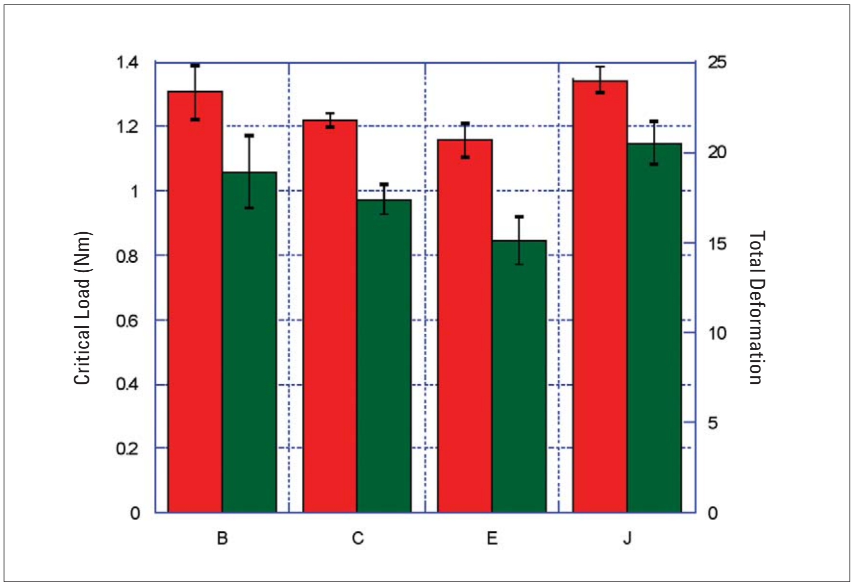

Figure 8. *Critical Load* and *Total Deformation* for the four top performing samples (based on *Critical Load*). Ideally, the results for *Critical Load* will be high and the *Total Deformation* will be low.

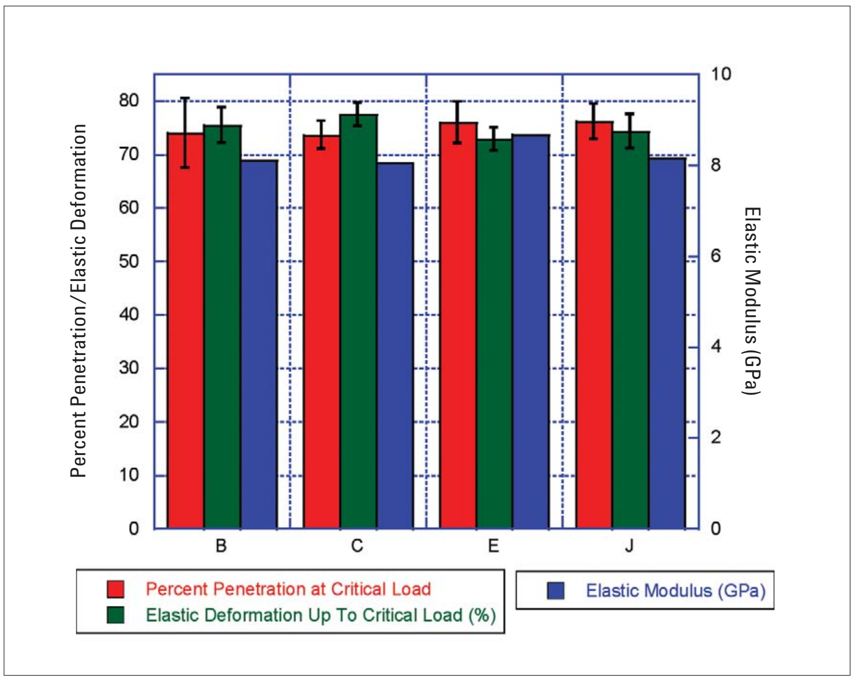

Figure 9. *Percent Penetration/Elastic Deformation* and *Elastic Modulus* for the four top performing samples (based on *Critical Load*).

	Film Thickness	Elastic Modulus	Hardness	Critical Load	Total Deformation	% Penetration
Film Thickness		1%	1%	67%	72%	59%
Critical Load	67%	3%	18%		97%	82%
Total Deformation	72%	3%	15%	97%		86%
% Penetration	59%	5%	17%	82%	86%	
% Elastic Deformation	43%	21%	10%	11%	15%	15%

Table 4. Correlation table of results.

- Very weak ≥40%
- Weak ≥50%
- Moderate ≥60%
- Strong ≥75%

values. There is insufficient information to conclude whether the solvent or film thickness is the culprit for the lower *Critical Load* value in Sample E. Given that the *Percent Penetration* in Figure 9 is the same for both samples E and J would suggest that the film thickness is the main cause; however, because different solvents were used in application, a final conclusion can not be drawn for the direct cause of the lower critical load. These two samples did show statistical differences in the results for elastic modulus confirming that the films have slightly different mechanical properties.

Correlation of Results

It is easy to look at a large matrix of test results, focus on large numerical differences in single columns, and overlook significant independent results. By examining the correlation of results, patterns can be recognized ensuring that the sample results are analyzed based on independent results instead of on groups of results that have strong correlations to a single parameter. A good example of a strong linear correlation is in Figure 8 where there is obviously a strong correlation between the *Critical Load* and the *Total Deformation*; if a researcher decided to neglect other information and focus on these two parameters as a basis for analyzing the performance of these films, both of these results will draw the same conclusion. Many times correlations are not as easily seen as demonstrated in Figure 8 — this one is easily recognized because there happens to be a 97% linear correlation between these two results — it is when the correlation drops below 60% that they become hard to recognize. For analyzing the correlation of the results in these scratch tests, normal correlation analysis was conducted and is described elsewhere [2]. In order to rate the levels of correlation between results, four levels were defined: very weak correlation (40 to 50%), weak correlation (50 to 60%), moderate correlation (60 to 75%), and strong correlation (>75%).

Table 4 lists the correlation levels between different results provided for the low-k samples.

It is apparent from the correlation analysis that all of the results — save hardness and elastic modulus which were measured using a test method specifically developed for measuring substrate independent properties — are, at least, weakly dependent on film thickness. The variation in *Critical Load*, for example, can be accounted for by a linear relationship with *Film Thickness* approximately 67% of the time. However, these result should not be neglected base on this correlation, because this is only a moderate correlation, with Sample E showing an obvious exception; Sample E is one of the thinnest samples but also had one of the highest critical loads. Some of the correlations shown in the table are of no surprise, such as *Percent Penetration* and *Total Deformation* coming in with a correlation of 86%; higher penetration at film failure would logically yield in higher *Total Deformations*. In fact, if the *Total Deformation* is correlated to *Penetration Depth at Critical Load*, as opposed to *Percent Penetration* which is normalized by the film thickness, the correlation factor jumps to over 98%. Some of the more surprising results were the almost complete absences of correlations to results of hardness, elastic modulus, and *Percent Elastic Deformation*. It is speculated that a correlation to hardness is absent because the interface between the film and substrate failed prior to failure of the film itself.

Conclusions

As a standard test method on the Agilent Nano Indenter platform, ramp-load scratch testing was used for evaluating the scratch response of low-k films on silicon substrates. All of the low-k samples tested experienced blistering of the film well before complete film failure occurred and imaging was completed, using the Nano Vision option, for confirmation of the mode of failure.

In addition to providing significant statistical differences in the results of the scratch tests, the results themselves were analyzed using correlation analysis. All of the scratch results were found to at least have a very weak correlation to film thickness. This weak dependence on film thickness makes intuitive sense because in usual scratch tests the film does not fail until the probe has significantly penetrated the film. Hence, the stresses from the scratch test propagate well into the substrate. There were also some surprising results of complete absence of correlation. Neither hardness nor elastic modulus correlated to any of the scratch parameters, and the *Percent of Elastic Deformation up to the Critical Load* did not correlate to any result other than *Film Thickness* — which was only a very weak correlation.

Sample J was determined to be the best performing sample due to its excellent ability to resist deformation, withstand a high percentage of penetration, and support the highest load before failure. Even though the results on Sample J were very similar to Sample B, Sample J was selected because the tests contained a lower standard deviation proving that it was more repeatable and predictable.

Agilent Technologies would like to thank SBA Materials for supplying the samples and some of the results for this study.

References

1. Hay, J., *Measuring substrate-independent modulus of dielectric films by instrumented indentation.* Journal of Materials Research, March 2009, Volume 24, pp.667–677.
2. Freund, John E., *Mathematical Statistics,* 5th Edition, New York: Prentice Hall, 1992.

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