NANOMECHANICS, INC.

Analytical Services Laboratory
105 Meco Lane
Suite 100
Oak Ridge, TN 37830
(865) 978-6490 Ext. 300

ServiceLab@nanomechanicsinc.com

When testing soft films on hard substrates should I report the results for the minimum hardness and elastic modulus properties or the hardness and substrate-independent elastic modulus results at 10% of the film thickness using the Hay-Crawford analysis?

Samples

Four samples were used to demonstrate the indentation results provided from the Thin Films test method used in the Nanomechanics' Analytical Services Laboratory. Data from these samples are used to clarify the results from the test method. The samples were labeled 1 through 4 with respective film thicknesses of 547.4 nm, 400.9 nm, 536.8 nm, and 580.9 nm. The samples are shown mounted to a 1.25" sample stub in Figure 1.

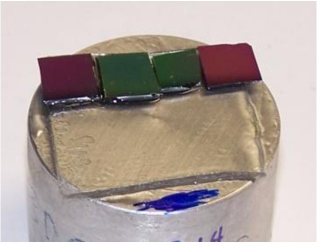

Figure 1: 4 wafer samples mounted and ready for testing.

Test Protocol

Testing was completed on the Nano Indenter ® G200 in the Nanomechanics' Analytical Services Laboratory using the Dynamic Contact Module (DCM) – the DCM is used for ultra-low force and ultra-high resolution indentation tests – with a Berkovich shaped indentation tip. Dynamic indentation was completed using the Continuous Stiffness Measurement (CSM) technique which allows the evolution of mechanical properties to be observed as the indentation tip penetrates the film at a constant indentation strain rate – the CSM technique is further described elsewhere [1]. Nanomechanics' has optimized this test technique to allow high throughput and unmatched accuracy by leveraging our expertise in control and data acquisition and carefully tuning the software controls of the Nano Indenter® G200.

The analysis of the data followed the standard data analysis purposed by Oliver and Pharr [1]. Results for the minimum elastic modulus and hardness are reported along with the Hardness measured at 10% of the film thickness. In addition to this standard analysis, the Hay-Crawford analysis technique was used to determine and report the substrate-independent properties of elastic modulus at 10% of the film thickness. Traditionally the results of elastic modulus are heavily influenced by the substrate material at 10% of the film thickness; but, the Hay-Crawford technique has been shown to accurately provide measurements of the substrate-independent properties of elastic modulus up to 40% of the film thickness [2]. It should be noted that the measurement of hardness is not usually affected by the substrate material at 10% of the film thickness – the hardness measurement is affected by the plastic zone of the stress field during indentation and this plastic zone is confined to close proximity with the tip of the indenter. The measurement of elastic modulus, however, is affected by the elastic zone of the stress field, and this zone propagates a very long distance and is unconfined; thereby, causing the measurement of elastic modulus of the film to be heavily influenced by the substrate material unless the influence of the substrate material is accurately accounted for and removed by the analysis. The Hay-Crawford analysis allows for this accurate compensation and is described in detail elsewhere [2].

Table 1: Inputs for the thin films nanoindentation tests.

Strain Rate Target	0.1	1/s
Harmonic Displacement Target	1	nm
Poisson's Ratio, film	0.3	
Poisson's Ratio, substrate	0.2	
Young's Modulus, substrate	170	GPa
Frequency Target	75	Hz
Film Thickness	547.4; 400.9; 536.8; 580.9	nm

Results

The results of hardness and substrate-independent elastic modulus (using the Hay-Crawford analysis) for the four thin-film samples are provided in Table 2. Clear statistical differences in the results are present for each sample. These statistical differences are also present in the results for the minimum detected hardness and elastic modulus listed in Table 3. There are two primary differences in the results from these two tables: the properties reported in Table 2 are taken at 10% of the film thickness while the results reported in Table 3 are automatically chosen by the software as the minimum detected hardness and elastic modulus from the penetration curves (at penetration depths not less than 20 nm); and, secondly, the elastic modulus measurements in Table 2 have been analyzed using the Hay-Crawford analysis to provide substrate independent results, where Table 3 provides the uncorrected measurements analyzed using the Oliver-Pharr technique developed for bulk materials. The results show that the substrate-independent elastic modulus taken at 10% of the film thickness is lower than the uncorrected minimum elastic modulus reported in Table 3. The primary reason for this is due to the uncorrected results being affected by the underlying substrate even at the location of the minimum properties on the penetration curve.

Figure 2 graphically displays the results of hardness for the four thin film samples. Little difference is observed in the hardness measurement between the results taken at the minimum and at 10% penetration; while the results taken at 10% are slightly higher, the standard deviation in the results taken at the two different locations from the penetration curves cross in all but the thinnest film. It can be concluded that the hardness of the film is affected very little by the substrate material up to 10% of the film thickness. The one significant difference in these two results is the scatter in the results. Notice that the standard deviation for the results of hardness is cut in half when the results are taken at 10% of the film thickness. For this reason, it is recommended that the results for hardness taken at 10% of the film thickness be reported. Nanomechanics provides both results of hardness for historical tracking of measurements or for comparison to other test protocols that would provide only the results for the minimum properties.

Figure 3 shows the evolution of hardness as a function of normalized penetration into the film – the measurement of *Normalized Displacement into Surface* is calculated as the penetration depth divided by the film thickness and the result is displayed as a percentage. While there is a hardness drop at the surface of the film (0% for the normalized displacement into surface) this is at penetration depths that are only 10's of nanometers deep and where the tip shape is not as well defined. At depths greater than 5% the hardness remain relatively constant to about 18% penetration – except for the thinnest film where there may well be a hardness gradient of the film.

The results of elastic modulus at the two ranges show very different results. Figure 4 shows a bar graph of the results reported in tables 2 and 3; in addition; the uncorrected results of elastic modulus taken at 10% of the film thickness are also provided to show the raw measurement of elastic modulus with substrate influences. The standard deviations in the results do not cross on any set displayed; this is because the results for elastic modulus are a strongly affected by the substrate material as the film is penetrated. This dependence on penetration depth is clearly seen in Figure 5 where the uncorrected elastic modulus is displayed as a function of normalized penetration; notice that there is not a consistent range where the elastic modulus is constant. Once again the error bars in Figure 4 are much smaller for the results taken at the deeper penetration depth, but it is clear that the uncorrected results for elastic modulus taken at 10% are too heavily affected by the substrate to be of value in reporting.

Figure 6 shows the evolution of the substrate-independent measurements of elastic modulus as a function of normalized penetration; these curves shows no dependence on penetration depth up to approximately 40% of the of the film thickness due to the accurate modeling of the substrate influences in the Hay-Crawford model. It is much easier to examine differences in the results of elastic modulus for multiple samples throughout the range of penetration by examining the substrate-independent results displayed in Figure 6 as opposed to the uncorrected data displayed in Figure 5. Due to the insensitivity in the results of the substrate-independent elastic modulus as a function penetration depth and because of the lower scatter in the results, it is recommended that the results for the elastic modulus of the film at 10% penetration be reported. Once again, Nanomechanics provides both results of elastic modulus for historical tracking of measurements or for comparison to other test protocols that would provide only the results for the minimum properties.

Two other reasons for choosing to report the results at 10% penetration using models that account for and remove the substrate influences include better tip area functions and less environmental noise. Manufacturing of the calibrated indentation tips has become far more consistent over the past 10 years, however, the smallest tip radii available for a Berkovich indentation tip is approximately 20 nm. Even with the higher reproducibility, the tip area functions have more variability over time at these length scales. Therefore, the area functions at 10% penetration are inherently more stable than at the apex of the tip. Secondly, any vibrations or thermal drift caused by environmental noise affects the data greater at near surface penetration depths. The results determined at 10% of the film thickness – using models that account for the substrate influence - are more stable, provide greater reproducibility among different instruments, and provide greater reproducibility over time.

Table 2: Results for the elastic modulus of the film and hardness at 10% of the film thickness; the standard deviations for each results is also provided.

Sample	Elastic Modulus _ Film (9.5 % to 10.5 %)	E _ Film St Dev.	Hardness (9.5 % to 10.5 %)	H _ 10% St Dev.
	GPa		GPa	
1	3.89	0.03	0.61	0.01
2	5.11	0.06	0.77	0.01
3	3.71	0.06	0.55	0.01
4	4.32	0.03	0.67	0.01

Table 3: Results for the measured minimum elastic modulus (without correction for substrate influences) and minimum hardness; the standard deviations for each results is also provided.

Sample	Minimum Apparent Modulus	Min E St Dev.	Minimum Hardness	Min H St Dev.	Depth At Min E	Depth At Min H
	GPa		GPa		Angstrom	Angstrom
1	4.33	0.07	0.58	0.02	243	237
2	5.67	0.13	0.71	0.03	226	211
3	4.19	0.13	0.53	0.02	244	563
4	4.72	0.09	0.64	0.02	223	268

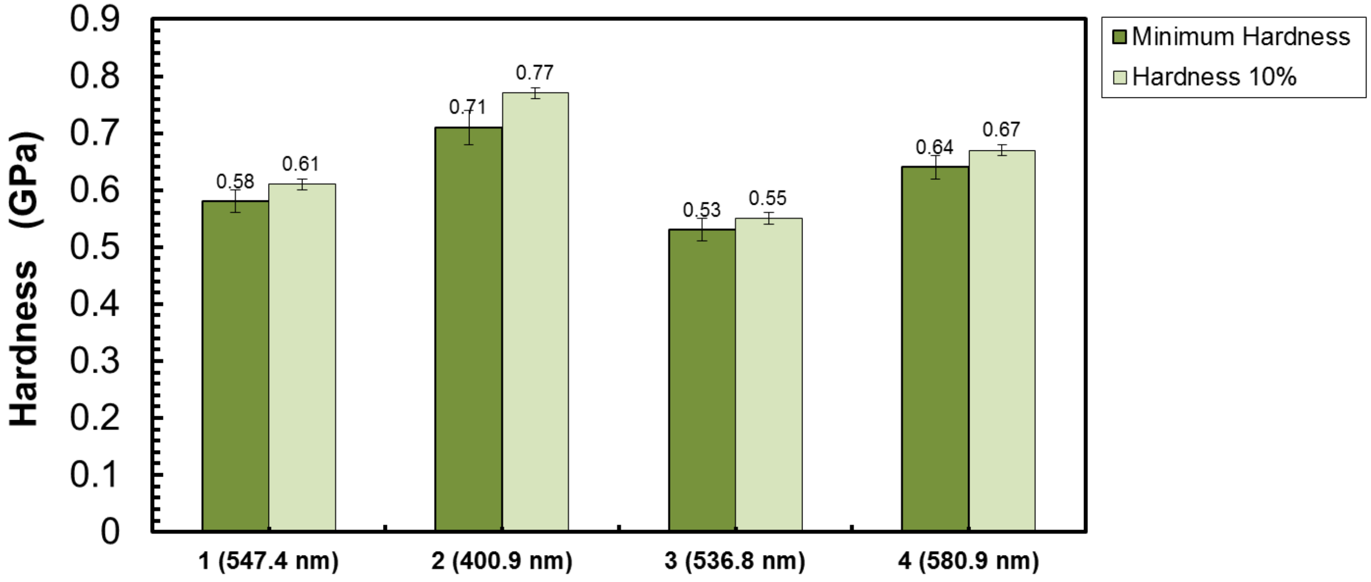

Figure 2: Results for the minimum hardness compared to the results for the hardness at 10% of the film thickness.

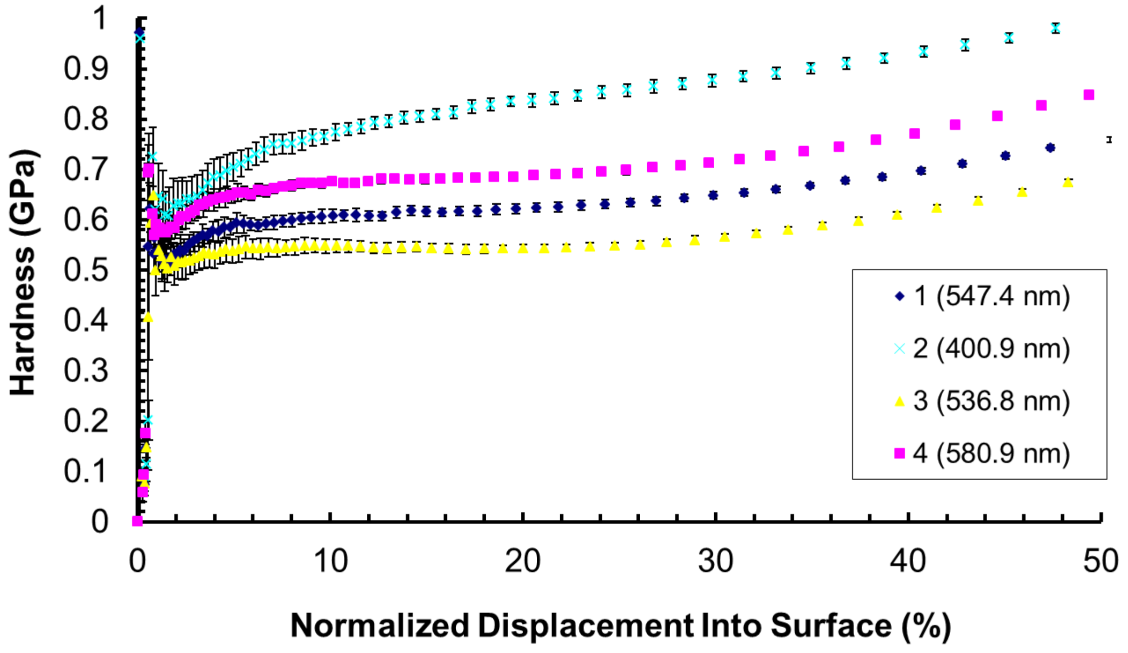

Figure 3: Evolution of hardness as the indenter penetrated the film.

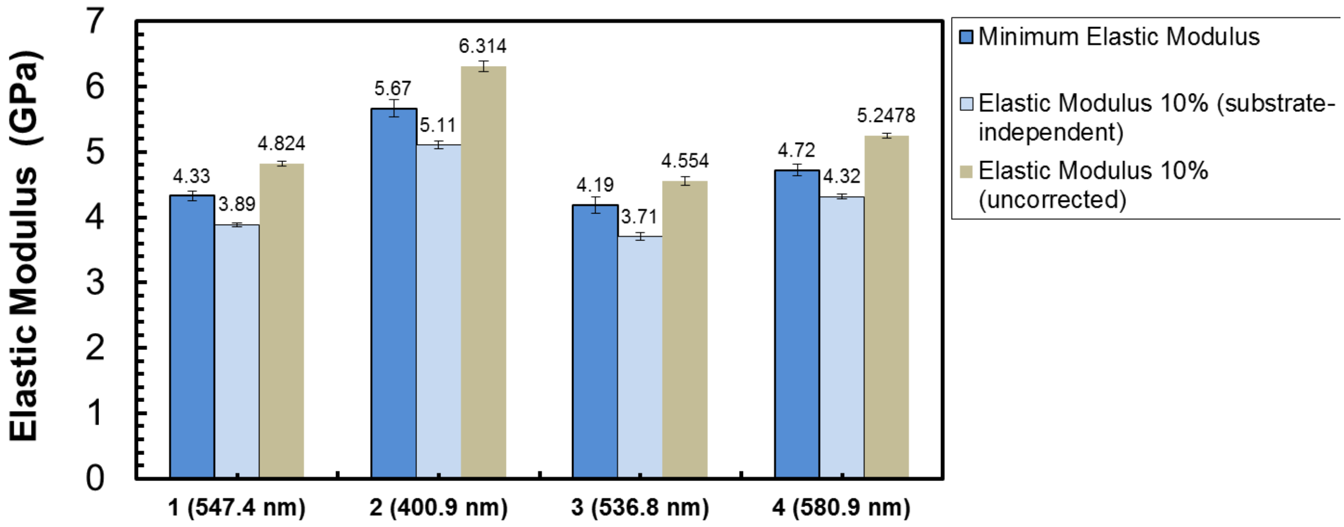

Figure 4: Results for the *minimum elastic modulus* as compared to the result for the *substrate-independent* elastic modulus and *uncorrected elastic modulus* at 10% of the film thickness.

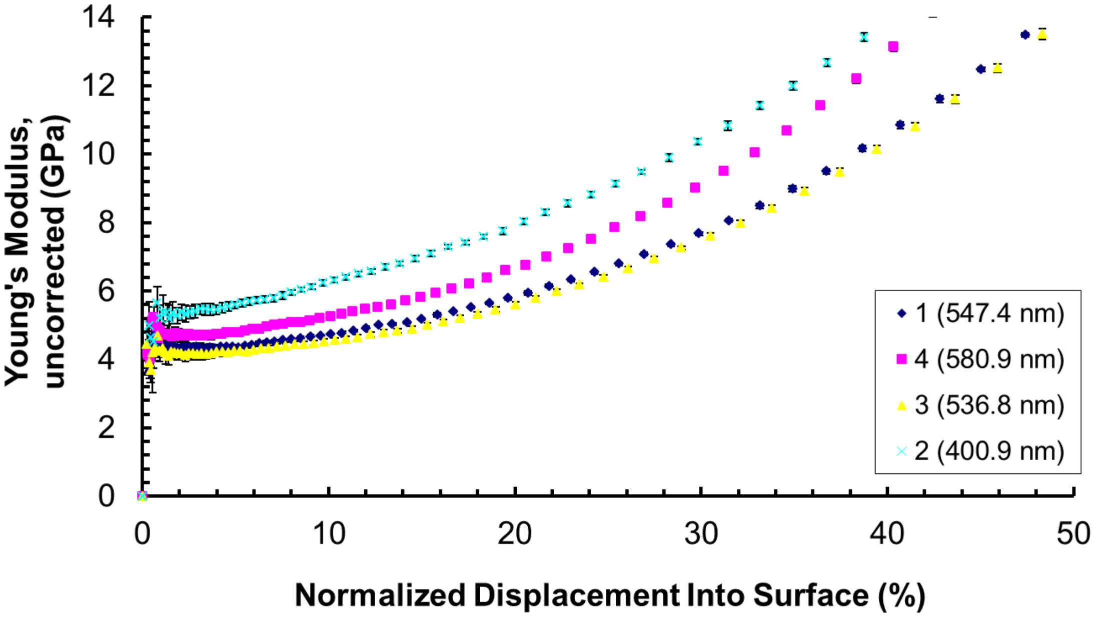

Figure 5: Evolution of elastic modulus (uncorrected results) as the indenter penetrated the film.

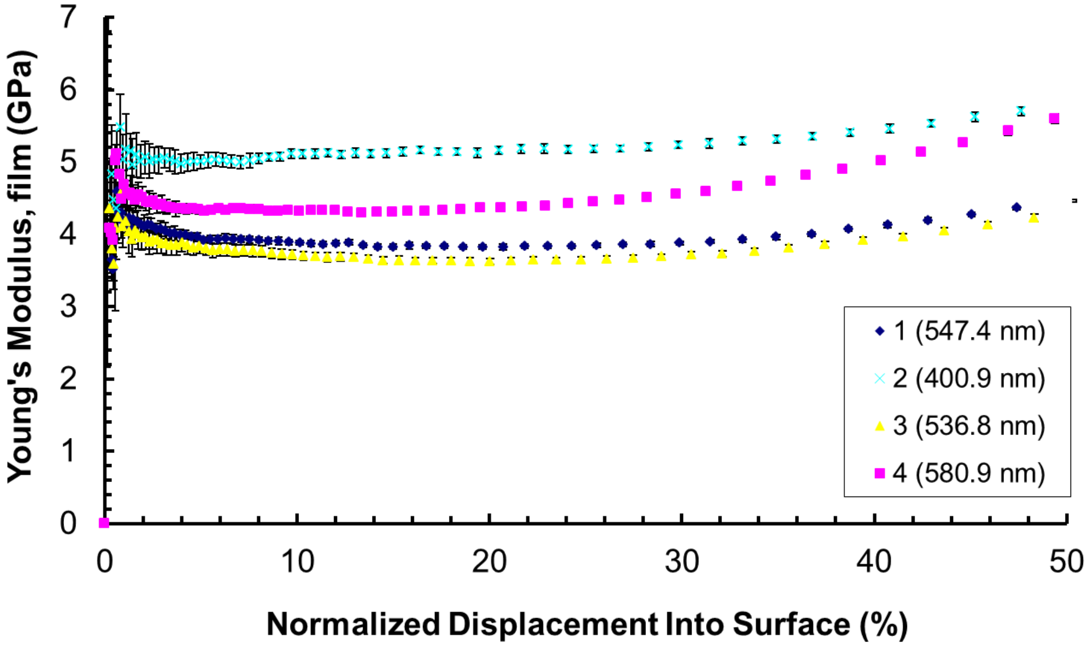

Figure 6: Evolution of elastic modulus (substrate-independent results) as the indenter penetrated the film.

References

[1] W.C. Oliver and G.M. Pharr, “Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology,” *J. Mater. Res.,* vol. 19 # 1 (2004).

[2] J.L. Hay, “A new model for measuring substrate-independent Young’s modulus of thin films by instrumented indentation,” Agilent Technologies application note (2010).