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Measuring the mechanical properties of ultra-thin low-k films and a review of common analysis techniques

Introduction

In the preparation of low-k films for mechanical evaluation, the films are often deposited thicker than the “as-used” thickness. This is to minimize the influence of the substrate on the measurements of elastic modulus and hardness which is caused by the indentation stress field propagating through the film into the substrate and inflating the resulting properties. This article evaluates three commonly used analysis techniques for determining the elastic modulus of these films, two analysis techniques for determining the hardness of the films, and, finally, identifies the best analysis procedures for reporting the results. The evaluation is based on the results from testing three sets of low-k films – ranging in mechanical properties covering a usual range of elastic modulus and hardness for low-k films – that consisted of three thicknesses of nominally the same film composition. The results clearly identify the best analysis techniques for hardness and elastic modulus of the films. The techniques used for the evaluation of the elastic modulus were: measuring the minimum properties based on the elastic modulus versus penetration curve, performing extrapolation based on the modulus versus penetration curve, and using a model that accounts for substrate influences to report the results at 10% of penetration into the film. Hardness was evaluated using two techniques; one technique reported the minimum value from the hardness versus penetration curve, while the other technique reported the hardness at 10% of penetration into the film. It is also shown that appropriate results can be provided on films in the “as-used” thickness range.

Samples

A new class of spin-on low-k films was evaluated for this article. These films had dielectric constants between 2.0 and 2.3. Nine samples were used to demonstrate the common analytical techniques for dynamically measuring the elastic modulus and hardness. These nine samples were divided into three sets where each set of samples consisted of nominally identical chemical compositions and processing parameters, but the samples in each set were deposited to different film thicknesses. Table 1 lists the three sets and the film thickness of each sample. The samples are shown mounted on a 1.25” sample stub in Figure 1.

Table 1: Film thicknesses for the samples.

Sample Identification	Film Thickness
	nm
NMI 03 - 215 nm	215.1
NMI 03 - 361 nm	361.9
NMI 03 - 600 nm	600.1
NMI 02 - 195 nm	195.3
NMI 02 - 345 nm	345.6
NMI 02 - 584 nm	584.2
NMI 01 - 195 nm	195.2
NMI 01 - 362 nm	362.2
NMI 01 - 562 nm	562.2

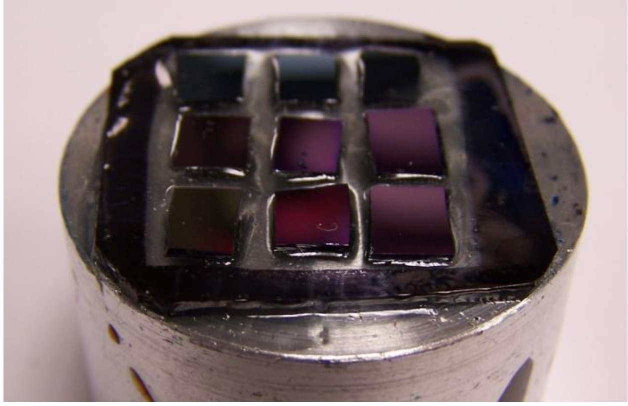

Figure 1: Nine wafer samples mounted and ready for testing.

Test Protocol and the Analysis Techniques

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 test protocol did not differ for any of the measurement techniques – only the post analysis of the data changed to examine the differences in the results due to analysis techniques. The input test parameters for the tests are listed in Table 2 and each test progressed as follows,

1. The tip approached the surface of the sample until contact was detected.
2. Loading was conducted at a constant indentation strain-rate while maintaining a 1 nm dynamic oscillation for the CSM technique until the penetration depth exceeded 50% of the film thickness.
3. The peak load was held constant for 10 seconds.
4. 90% of the force was withdrawn at a controlled rate.
5. The thermal drift of the sample was evaluated for a period of 75 seconds.
6. Finally, the sample was unloaded completely.

The results from the test provided the continuous measurements of Stiffness, Load, and Displacement as the tip penetrated the film. These data further provided continuous measurements of contact area, elastic modulus, and hardness. Traditionally, the measurements of elastic modulus and hardness are determined using the Oliver-Pharr analysis and are not compensated for any substrate influence. In this analysis, the measurements of stiffness and contact area are used to determine the reduced elastic modulus, E_r - the reduced elastic modulus includes an elastic component of the indenter tip material in addition to the measured materials response. Equation 1 is used to determine the reduced elastic modulus.

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{1}$$

Where β is a geometric constant that accounts for using a pyramidal tip geometry that deviates from an axisymmetric geometry, such as a cone; β is typically taken to be 1. Since the indenter material is well known, typically diamond, the response of the tip can easily be removed using Equation 2, which provides the elastic modulus.

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{2}$$

Where E is the elastic modulus of the sample, E_i is the elastic modulus of the indenter tip, ν is the Poisson's ratio of the sample, and ν_i is the Poisson's ratio of the indenter.

Two results for the elastic modulus were reported using the elastic modulus as determined from Equation 2. First, the minimum elastic modulus from the elastic modulus versus penetration curve was reported. Secondly, the extrapolation technique outlined by ISO 14577 was used to determine the elastic modulus of the film. This technique states that data collected below the penetration depths where $\frac{a}{t_f} < 1.5$, a is the contact radius and t_f is the thickness of the film, are used to extrapolate the data back to zero penetration and this value is reported. In doing this, data collected between 5% and 10% of the thickness of the film – for a sharp Berkovich shaped indenter tip this correlates to $\frac{a}{t_f} < 0.25$ – was used to perform a linear fit and extrapolated the data back to zero penetration.

One additional analysis technique was used to determine the elastic modulus of the sample; this technique was purposed by Hay *et al* [2]. In this technique the user enters the film thickness of the sample, Poisson's ratio of the film (0.3 is commonly used for low-k films), and the substrate properties (elastic modulus and Poisson's ratio). The film and substrate are then modeled in such a way that allows the contribution of the film and substrate properties to vary as the test progresses and the indenter penetrates further into the material. The contributed amount of the film and substrate properties to the measured apparent shear modulus is defined by a weighting function, described in further detail elsewhere [2]. By isolating the contribution of the film properties to the measured shear modulus, the elastic modulus of the film is determined using Equation 3.

$$E_f = 2\mu_f(1 + \nu_f) \qquad (3)$$

Where E_f is the elastic modulus of the film, μ_f is the shear modulus contribution of the film determined using a weighting function, and ν_f is the Poisson's ratio of the film.

The general rule of thumb for indentation states that the hardness can be measured up to 10% of the film thickness and not be overwhelmingly influenced by the substrate material [3]. To test this, values were reported for the hardness at 10% penetration and also for the minimum detected hardness on the hardness versus penetration curve to see if the same hardness value was measured for the different film thicknesses in each set.

Hardness of the sample was determined using Equation 4.

$$H = \frac{P}{A} \qquad (4)$$

Where, P is the load applied to the sample and A is the contact area.

Table 2: 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	See Table 1	nm

Results

Each sample was tested ten times and the results were averaged and over a small range of penetration and reported. The results of the hardness measurements are provided in Table 3. One of the hardness measurements provides a result of the averaged hardness over the range from 9.5% to 10.5% of penetration into the film, while the minimum hardness measurement used the hardness versus penetration curves to locate the minimum hardness present in the graph and average the results over a 5 nm range around the minima. Figure 2 displays the curves of the Hardness versus Normalized Displacement into Surface (percent penetration); data for each sample has been discretized over the penetration range and one standard deviation in the measurement is represented by the error bars on the graph. The advantage of the CSM technique is clearly present in this figure; without *a priori* knowledge of the response of the film, a test measures the complete evolution of the mechanical properties from the surface of the film into the substrate and a range of properties can be determined from a single test. A gradual rise in the hardness around, and after, 10% of film penetration is seen for all of the samples; this rise is caused by substrate influences in the results. Figure 3 displays the results from Table 3 in a bar graph format; from this figure it is clearly seen that the hardness results measured at 10% penetration does contain substrate influences because the results correlate directly to film thickness for each sample set. The minimum hardness values, though, do not correlate directly to the film thickness and these results show statistically the same hardness result for all of the samples in each set.

Table 4 lists the three results for the measurements of elastic modulus; Figure 4 shows the bar graph for these results. The substrate-independent measurements which were determined using the Hay-Crawford model show excellent ability to measure the elastic modulus of the samples independent of the film thickness; the results for the elastic modulus of the film at 10% penetration are statistically identical for all three film thickness in each sample set. This cannot be said for the results of the Minimum Elastic Modulus or the Extrapolated Modulus. These results show that the measurements of elastic moduli are affected by the substrate material even at penetration depths less than 5% of the film thickness; this is because the elastic stress field, from which the elastic modulus is determined, extends great distances into the sample. Even more, the extrapolate results have a large range for which the extrapolation can be performed and the results will vary greatly based on the selected range. The substrate influence is not seen in the minimum hardness results because hardness results are affected by the plastic stress field, which is confined to close proximity of the tip.

Conclusions

The mechanical properties of three sets of low-k films have been measured where each set consisted of nominally the same film deposited to different film thicknesses. Three analysis techniques were explored for reporting the elastic modulus of the films and two analysis techniques were explored for reporting the hardness of the films. It was found that the only technique that provided consistent results of elastic modulus on the different film thicknesses was the Hay-Crawford analysis. The techniques of picking the minimum elastic modulus from the *Elastic Modulus* versus *Penetration* curve and using the elastic modulus curves to extrapolate the data back to zero penetration showed strong correlations to film thickness and neither provided consistent results for the nominally identical film compositions of different thicknesses. In measuring the hardness, the minimum hardness measurements did not show a correlation to film thickness and these results provided the same value for the same films of different thicknesses. The measurement for the hardness at 10% penetration, though, did show a correlation to the film thickness.

Based on this set of data, it is recommended that the Hay-Crawford analysis be used for reporting the results of elastic moduli and the minimum measured hardness values be reported for the hardness when testing low-k films on silicon substrates. These two measurements provide the best known method for the determination of the mechanical properties when testing low-k samples that have varying film thicknesses. If the other techniques that are presented in this article – extrapolation, minimum modulus, and hardness at 10% - are used for reporting results, it will be difficult, or nearly impossible, to quantify how small changes in processing causes changes in the mechanical performance of the films if the thicknesses are allowed to vary.

Table 3: Averaged results for the hardness at 10% of the film thickness and the minimum measured hardness; one standard deviation in the measurement of each result is also provided.

Test	Hardness (9.5 % to 10.5 %)	H_StDev	Minimum Hardness	Min Hardness StDev
	GPa	GPa	GPa	GPa
NMI 03 - 215 nm	1.13	0.03	1.13	0.04
NMI 03 - 361 nm	1.19	0.03	1.15	0.06
NMI 03 - 600 nm	1.19	0.03	1.15	0.06
NMI 02 - 195 nm	1.03	0.06	1.05	0.06
NMI 02 - 345 nm	1.12	0.06	1.06	0.06
NMI 02 - 584 nm	1.14	0.01	1.01	0.04
NMI 01 - 195 nm	0.68	0.03	0.69	0.03
NMI 01 - 362 nm	0.7	0.03	0.69	0.03
NMI 01 - 562 nm	0.73	0.02	0.71	0.03

Table 4: Results for the measured minimum elastic modulus (without correction for substrate influences) and minimum hardness; one standard deviation in the measurement for each result is also provided.

Test	Elastic Modulus _ Film (9.5 % to 10.5 %)	E_Film StDev	Minimum Apparent Modulus	Min E StDev	Extrapolated Elastic Modulus	Extrapolated E StDev
	GPa	GPa	GPa	GPa	GPa	GPa
NMI 03 - 215 nm	7.72	0.16	9.77	0.23	9.18	0.41
NMI 03 - 361 nm	7.66	0.15	8.99	0.25	8.26	0.28
NMI 03 - 600 nm	7.52	0.11	8.27	0.25	7.52	0.27
NMI 02 - 195 nm	7.05	0.26	9.06	0.32	8.52	0.69
NMI 02 - 345 nm	6.88	0.25	7.99	0.26	7.31	0.26
NMI 02 - 584 nm	6.88	0.07	7.39	0.13	6.51	0.2
NMI 01 - 195 nm	4.41	0.11	5.67	0.13	5.9	0.36
NMI 01 - 362 nm	4.25	0.1	5.01	0.09	4.68	0.13
NMI 01 - 562 nm	4.34	0.07	4.9	0.1	4.44	0.11

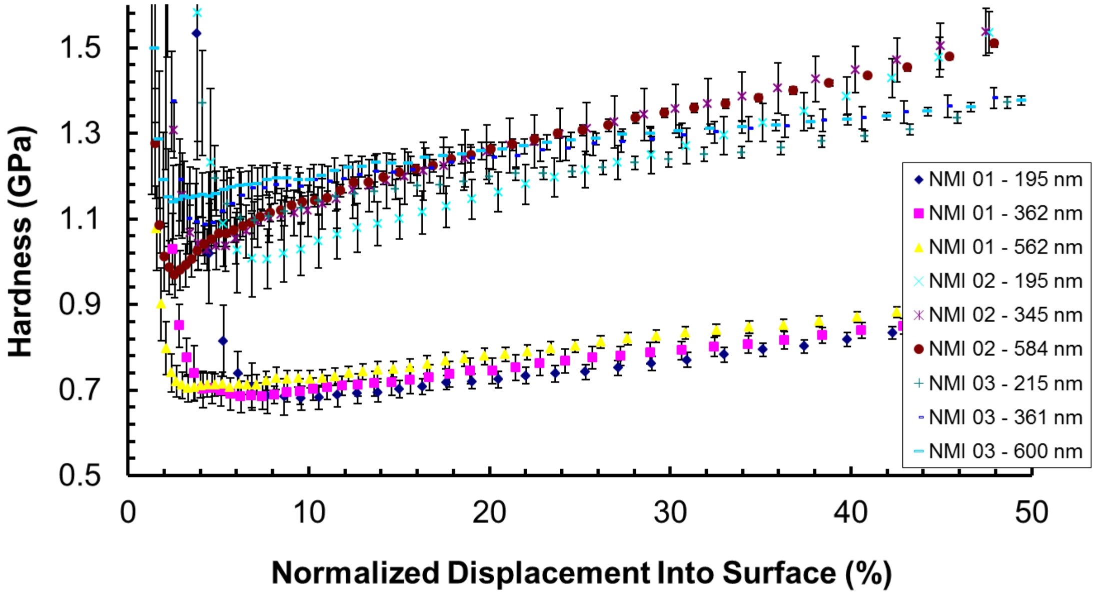

Figure 2: The results of hardness as the indentation tip penetrated through the sample; clearly, the results are being affected by the substrate material after 10% of penetration.

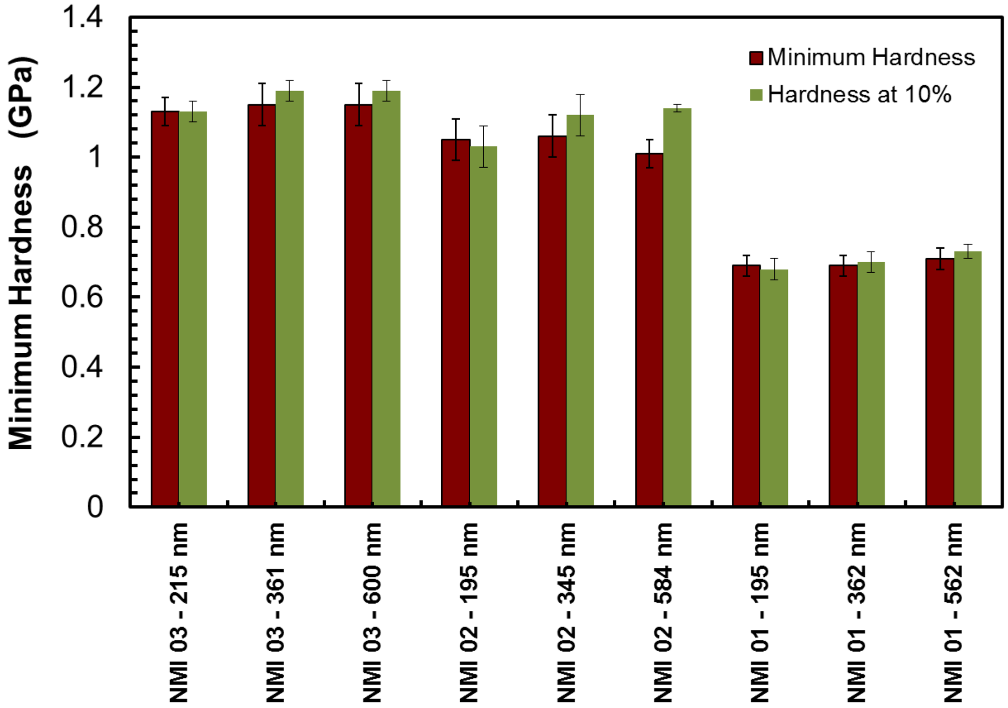

Figure 3: Results for the *minimum hardness* compared to the results for the *hardness at 10%* of the film thickness. The standard deviations in each measurement cross the mean of the other measurement, but the measurements at 10% of the film thickness show a direct correlation to film thickness while the minimum hardness results do not.

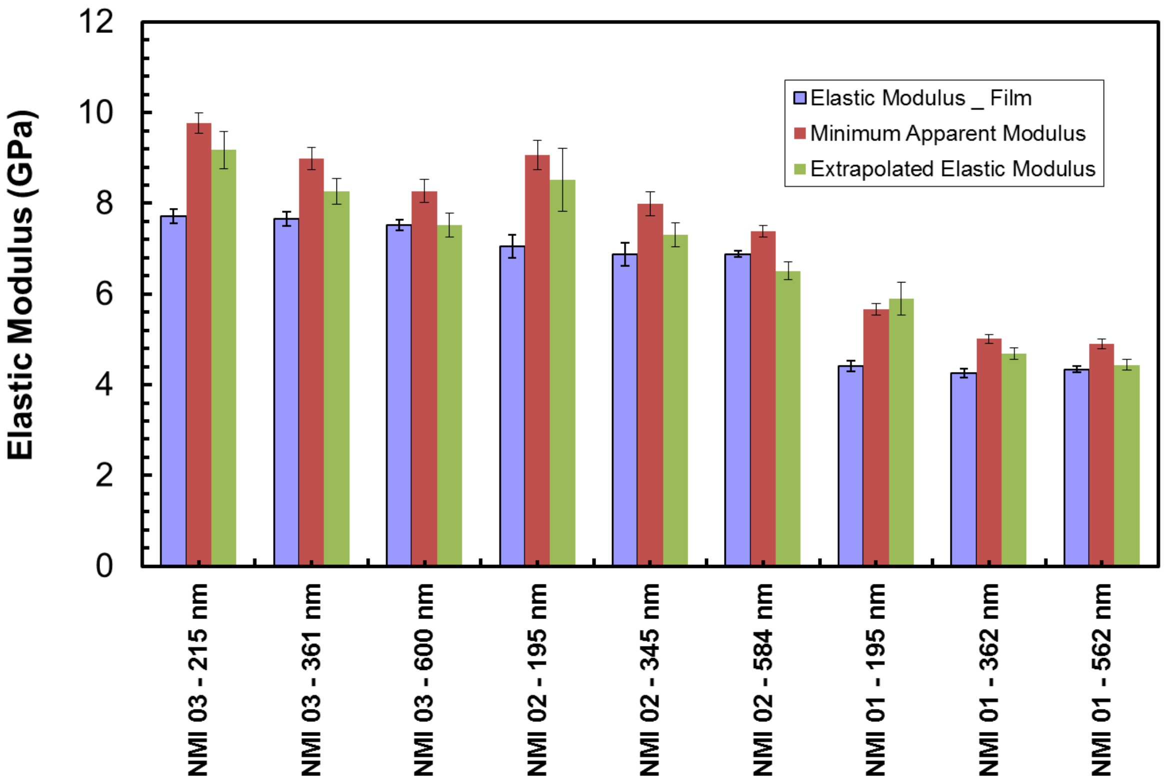

Figure 4: Results for the *elastic modulus of the film (substrate-independent)* as compared to the result for the *minimum apparent* elastic modulus (uncorrected) and *extrapolated elastic modulus*. The substrate-independent results are the only results that do not show a strong dependence on film thickness.

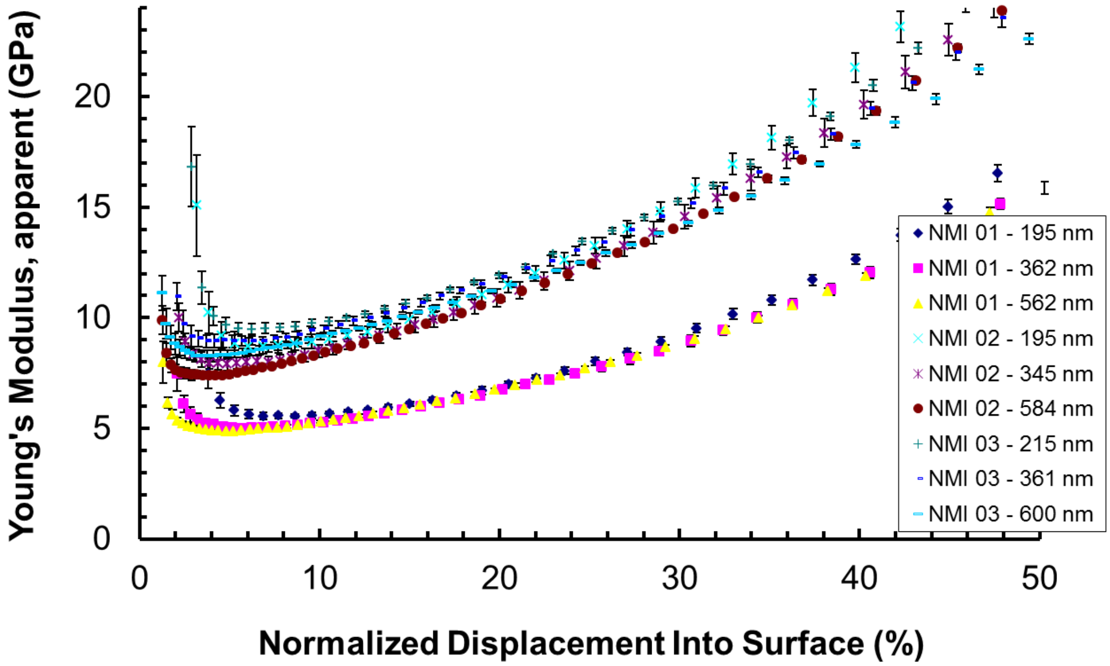

Figure 5: Evolution of the elastic modulus (uncorrected results) as the indenter penetrated the film. The minimums on these curves vary from 3% to 7% of the film thickness; this makes it difficult to determine the appropriate range for reporting the results.

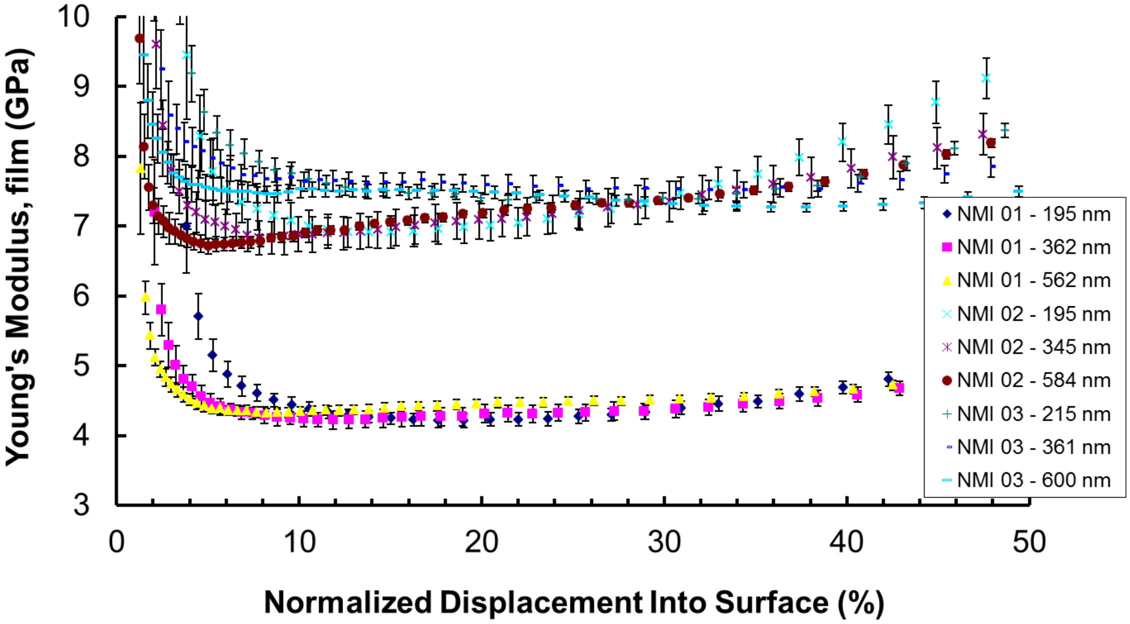

Figure 6: Evolution of the elastic modulus of the film (substrate-independent results) as the indenter penetrated the film.

References

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[2] J.L. Hay, B.A. Crawford, “Measuring substrate-independent modulus of thin films,” *J. Mater. Res.,* vol. 26, # 6 (2011).

[3] G.M. Pharr, J.L. Hay, “Instrumented Indentation Testing (IIT),” *ASM Handbook - Mechanical Testing and Evaluation*, vol. 8, pg. 232-243, (2000).