# Indentation *Rules of Thumb*

## — Applications and Limits

### Application Note

## Introduction

A wealth of information can be derived from instrumented indentation testing (also called nanoindentation); this testing provides the necessary data for the determination of Young's modulus, hardness, elastic/plastic work, and other material properties. Over the years a set of generalized rules have been empirically determined including the *10% Rule* for indentation depth on coatings, the *5% Rule* for surface roughness, and the *1 Degree Rule* for surface tilt and alignment. This article addresses each of these rules and provides justification and limitations for each.

## Samples

Five samples were used to evaluate the three *Rules of Thumb*: smooth glass, rough M42 tool steel, 978 nm low-k film on silicon, 1.5 µm gold film on silicon, and an approximately 1 µm TiN coating on steel. This sample set represented bulk samples with varying surface roughness, soft films on hard substrates, and hard films on soft substrates.

## Test Procedure

Each *Rule of Thumb* was tested individually using samples that would best show the applicability and limits of the rule — the film samples were used for testing the *10% Rule*, the bulk samples without coatings were used for testing the *5% Rule*, and the smooth glass was used for testing the *1 Degree Rule*. All indentation tests were conducted on the Agilent Nano Indenter G200 using the Continuous Stiffness Measurement (CSM) option which allows dynamic indentation for the observation of mechanical properties as a function of penetration into the surface of the sample. The CSM option allows surface properties and bulk sample properties to be measured from a single indentation test.

In testing the *1 Degree Rule*, smooth glass slides were mounted on sample pucks that had been modified to tilt the sample at 0, 1, 3, 5, and 9 degrees. The angles were held within ±0.2° of the nominal angle when the samples were mounted in the sample tray — 8 mm single line scan were used to verify alignment.

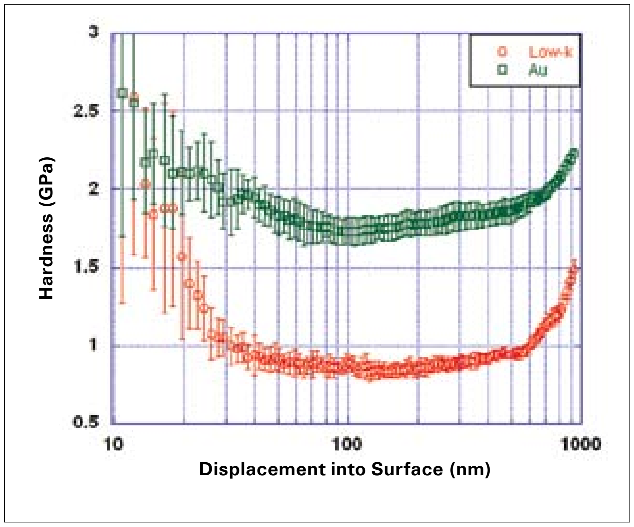


Figure 1. Hardness versus displacement into surface for the low-k (red) and the Au (green) films on silicon substrates. The film thicknesses of the low-k film was 978 nm and the Au film was 1.5 µm. These data are plotted on a semi-log plot for closer examination of the minima and plateaus associated with the curves.

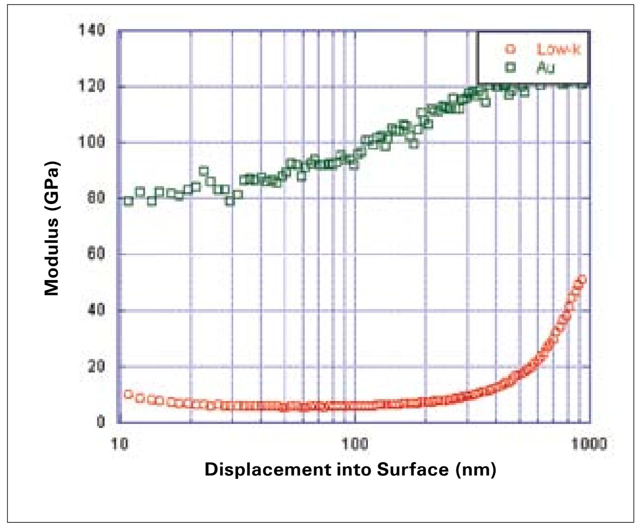


Figure 2. Elastic modulus versus displacement into surface for the low-k (red) and the Au (green) films on silicon substrates. These data are plotted on a semi-log plot.

## Results and Discussion

### The 10% Rule

The *10% Rule* refers to the maximum penetration depth to which an indentation test can produce substrate independent hardness measurements. Often, researchers will forego the limitation and apply the rule as a general limitation for all mechanical properties including Young's modulus [1]; substrate independent measurements of hardness can generally be taken at deeper penetration depths than measurements of elastic modulus because the zone of plasticity is much smaller than the elastic zone. Three materials were tested to examine the applicability of the *10% Rule*: a 978 nm thick low-k film on a silicon substrate, 1.5 µm gold film on silicon, and an approximately 1 µm TiN layer on steel. These samples were chosen to represent film/substrate combinations that are regularly seen in testing – soft films on hard substrates and hard films on soft substrates.

The first two samples tested represent soft films on hard substrates. With these film samples, two primary concerns are prevalent: substrate effects and pile-up. The *Rule of Thumb* says that substrate independent measurements of hardness can be made up to 10% of penetration into the film. To test this, the Au and low-k films were tested to 1 micron of penetration and the mechanical properties data were examined for plateaus under the 10% region.

Figures 1 and 2 display the results for the hardness and elastic modulus, respectively, for the Au and low-k films. The hardness results for the gold sample show that a short plateau is observed up to approximately 150 nm of penetration. The hardness results fit well with the *Rule of Thumb* for this particular sample. However, the same can not be concluded for the measurements of elastic modulus. Figure 2 shows that the elastic modulus of the Au film was measured correctly as compared to its nominal value (78 GPa) up to 1% of the film thickness; then, past the 1% mark, the modulus continuously increased and never exhibited a plateau. This indicates that the elastic stress fields propagated to the substrate shortly after contact with the surface of the sample. In this circumstance a model such as described by Rar, et al. should be used to analyze the indentation data and compensate for substrate influences [4].

Observed results on the Au film also showed high values for hardness; the nominal values for the elastic modulus and yield stress of gold are approximately 78 GPa and 205MPa, respectively [5]. Using Tabor's approximation that the yield stress is equal to the hardness divided by three shows that the hardness value measured here is almost 3 times too high [6]. High hardness values are commonly caused by two phenomena other than substrate influences: indentation size effect and/or pile-up. To observe the causes of the separation in measured and nominal properties, one of the indentations was imaged using the Nano Vision® option. The Nano Vision option allows imaging to be conducted on a Nano Indenter system through the use of a high-precision piezo 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.

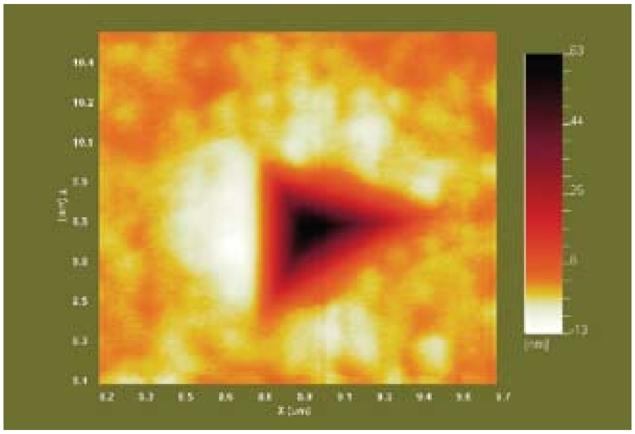

Figure 3. Nano Vision scan of the Au film after indentation to 100 nm. Only 13 nm of maximum pile-up is observed.

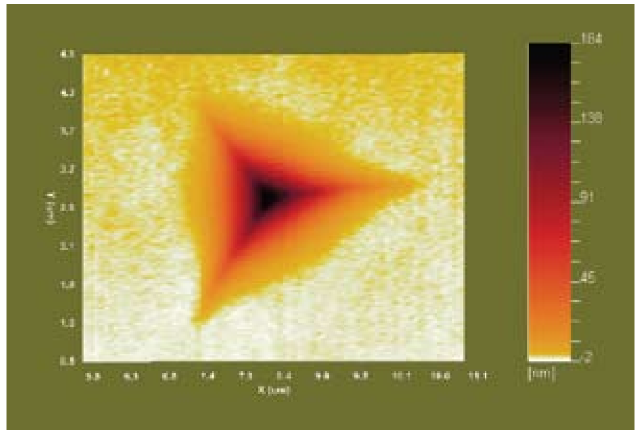

Figure 4. Nano Vision scan of the low-k film after indentation to 500 nm. Pile-up is not evident for this film.

Figure 3 displays the scanned image of a 100 nm deep indentation performed on the Au film. In this figure a small amount of pile-up is observed around the impression but this is not enough to account for a 3X increase in hardness. Pile-up causes an overestimation of the mechanical properties because the contact areas are calculated using the Oliver-Pharr method assuming elastic contact theory which predicts sink-in not pile-up [2, 7]. Therefore, it is suspected that the inflated hardness values are due to a combination of pile-up and indentation size effect.

Pile-up is not apparent for the low-k film shown in Figure 4; this sample was scanned for comparison to the Au sample. The hardness results for the low-k film, displayed in Figure 1, shows a long plateau after approximately 60 nm of penetration following the "skin-effect" — the skin-effect on porous low-k materials is caused by the elimination of pores at the surface of the film during processing. In fact, this skin-effect probably constrains the material so that pile-up can not occur for this soft film/hard substrate system. The edge impressions of Figure 4 bow in and show a similar appearance to indentation shapes performed on materials that exhibit sink in (i.e. glasses and ceramics), while the faces of the impression bow out and show similarities to impressions in materials that pile-up (i.e. soft metals). The net effect is that no pile-up exists. The plateau region in Figure 1 extends to approximately 110 nm before substrate influences have a noticeable effect; in actuality, Hay has demonstrated that the data for thin porous low-k films (below 1 micron of thickness) are always under the influence of either the skin of the film or the substrate material — hardness much less so than modulus [8]. The elastic modulus for the low-k film, shown in Figure 2, also shows that the results are not heavily affected by the substrate until the penetration depth extends beyond 90nm of penetration. In the absence of the work completed by Hay, this would suggest that the *10% Rule* holds well for both the hardness and elastic modulus of low-k films.

The last film/substrate combination tested for application of the 10% rule was a hard film on a soft substrate. Figure 5 shows the results for hardness and elastic modulus on a TiN coating applied to M42 tool steel; the large scatter at shallow depths is indicative of high surface roughness. Similar to the Au sample, the hardness shows a plateau region but then sharply drops after a penetration depth of 70 nm. The curve for the elastic modulus of the TiN coating shows the complete absence of any plateau, which confirms that there is no region of the test where the elastic modulus is not affected by the underlying substrate. As this film is penetrated, the mechanical properties drop dramatically; which is expected since this is a hard film on a soft substrate. The exact film thickness of this coating is not known, but if the *10% Rule* is employed, then the approximate film thickness can be determined. In examining the response of the hardness curve, it is estimated that the thickness of the TiN layer is approximately 1 μm.

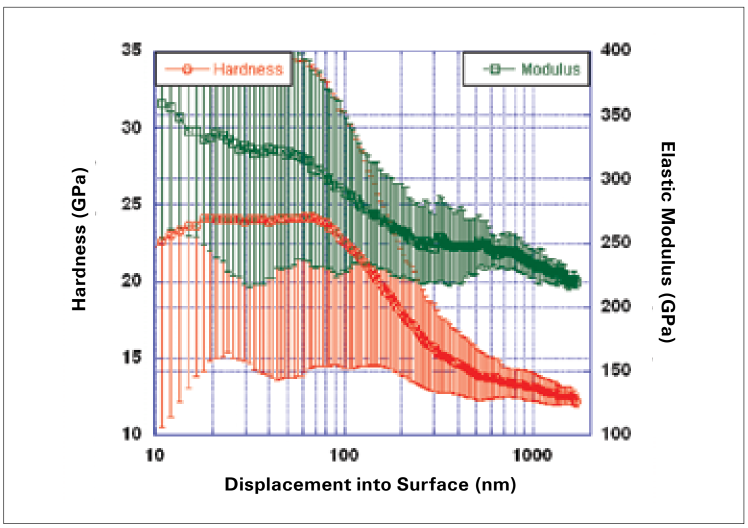


Figure 5. Hardness (red, left) and elastic modulus (green, right) versus displacement into surface for the TiN coating on tool steel.

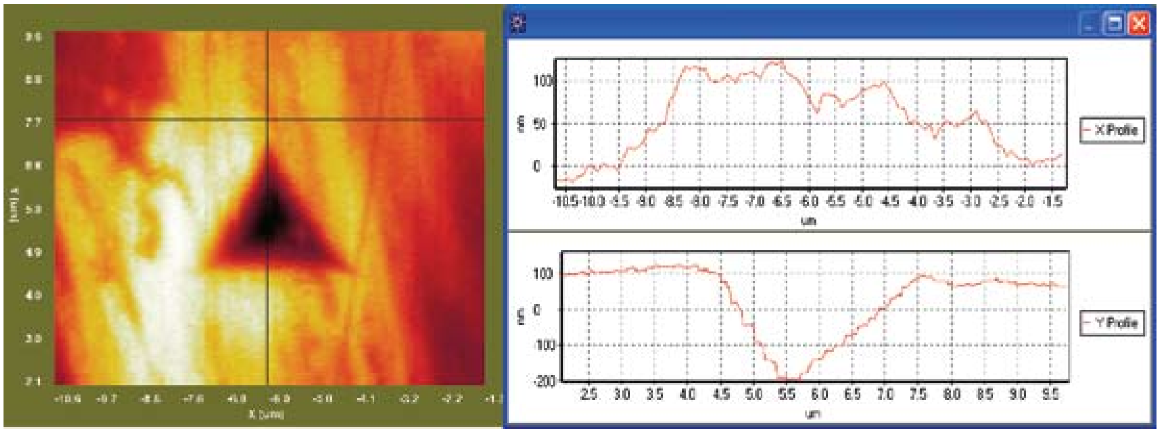


Figure 6. Nano Vision scan of the tool steel sample showing a surface roughness of 50 nm over a reasonable range for a Berkovich impression (approximately 5 µm).

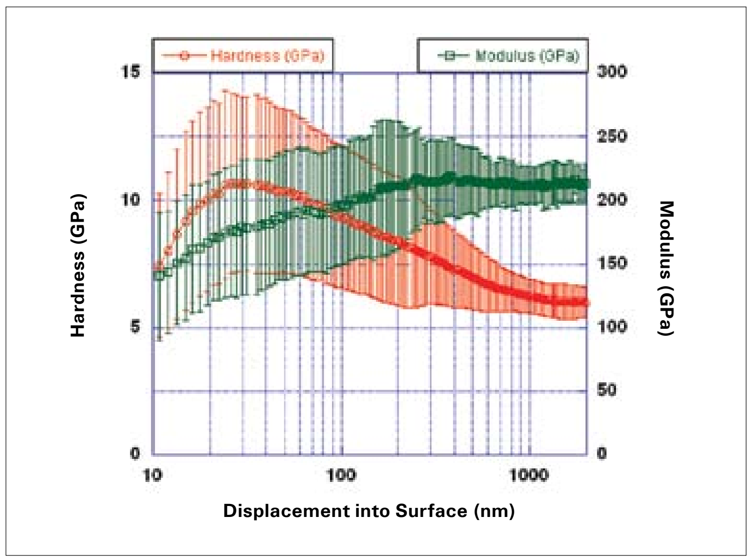


Figure 7. Hardness (red, left) and elastic modulus (green, right) versus displacement into surface for bare M42 tool steel.

**The 5% Rule**

Surface roughness is a challenge in instrumented indentation because it can lead to large errors in the contact areas that are used in determining mechanical properties. The greatest errors occur when the surface roughness is on the order of the contact dimensions [2]. The *Rule of Thumb* is that the surface roughness should be no more than 5% of the depth at which results are required. Therefore, to examine the applicability of this rule, rough tool steel and a smooth glass slide were tested to examine the penetration depths at which the data converged.

Figure 6 shows a scan of a 500 nm indentation made on the tool steel sample. This scan allowed for the measurement of the surface roughness and the estimation for the expected convergence of the mechanical properties. The X-Profile of Figure 6 shows approximately 50 nm of random surface roughness over a reasonable range for a Berkovich impression (approximately 5 µm in length). Using the *5% Rule* implies that an indentation depth of 1000 nm would be required for acceptable scatter in the results; an acceptable scatter in the results is usually defined as a covariance that is less than five percent. Figure 7 shows the results for the indentation tests on the tool steel sample. Indeed, the results at the surface of this sample show significant scatter, especially in the hardness results; hardness is affected more by surface roughness than the elastic modulus because of how the area terms are used in the calculation of these two properties. In the determination of hardness, the area term is used directly, while in the calculation of elastic modulus the square root of the area is used. The results speak for themselves. The elastic modulus is within an acceptable range at approximately 600 nm of penetration, but the hardness does not have an acceptable range of scatter until the penetration is over 900 nm.

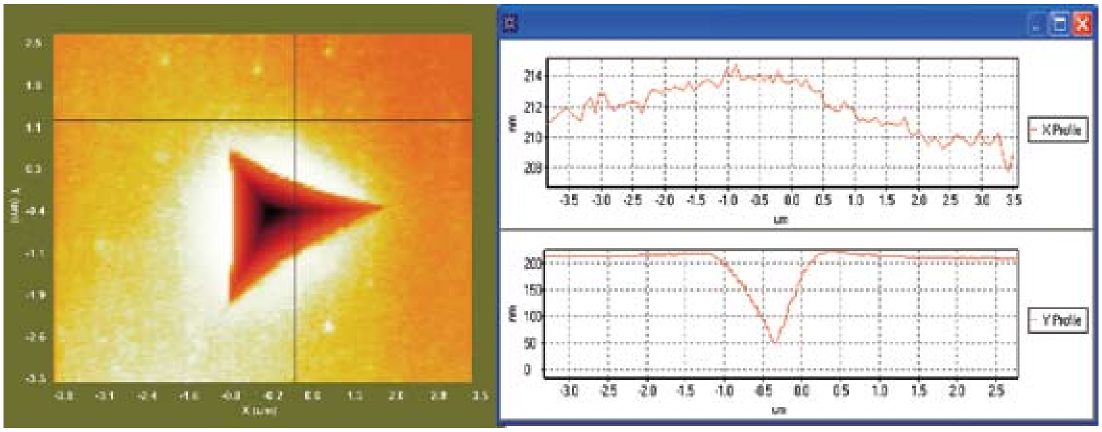


Figure 8. Nano Vision scan of the glass slide showing a surface roughness of 4 nm – over a reasonable range for a Berkovich impression (5 µm).

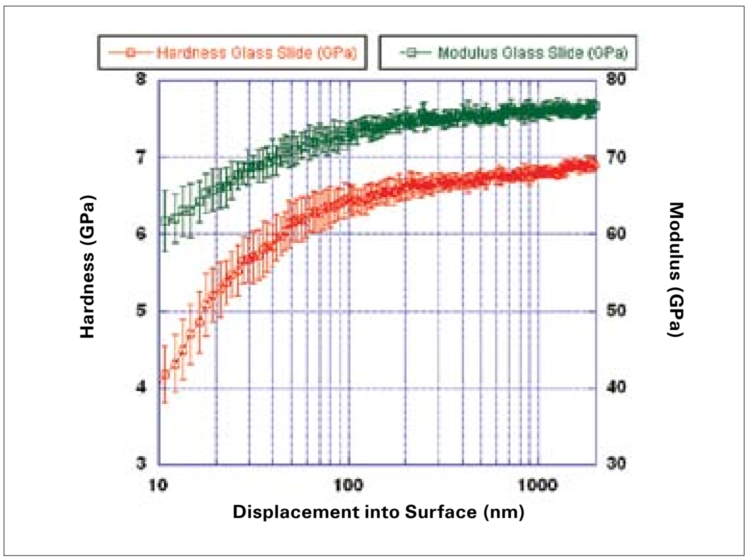


Figure 9. Hardness (red, left) and elastic modulus (green, right) versus displacement into surface for the glass slide sample.

The scan of the glass slide, displayed in Figure 8, shows that the surface roughness was approximately 4 nm over a reasonable range for a Berkovich tip impression. Using the *5% Rule* for this sample implies that mechanical properties could be reliably measured, with an acceptable range of scatter, at penetration depths greater than 80 nm. Figure 9, a plot of the hardness and elastic modulus results for the glass slide, confirms that the mechanical properties have a very low standard deviation above the limit described by this rule. Both, the results on the glass slide and the tool steel sample, show that quality measurements can be made on surfaces at penetration depths that are less than the minimum depth specified by the *5% Rule* — these measurements will just have a larger standard deviation associated with the results.

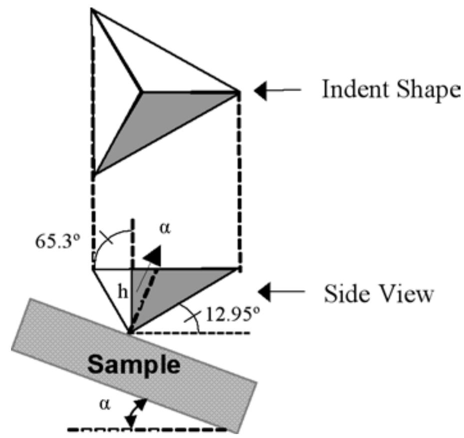


Figure 10. Diagram of an ideal Berkovich tip indenting a tilted sample.

**The 1 Degree Rule**

When testing surfaces that are not aligned orthogonal to the indenter, the determination of the contact area suffers along with possible lateral sliding of the contact. The *Rule of Thumb* for alignment is that the surface should be within one degree of orthogonal alignment with the tip — this is also stated as a requirement in the ISO standard for instrumented indentation testing [3]. To examine the errors caused by misalignment, analytical calculations were performed using an ideal Berkovich tip; then, glass slides with peak-to-peak random surface roughness of 1 nm and a surface roughness of 4 nm over a 5 μm range were mounted and tested on angled sample holders ranging from 0 to 9 degrees. The samples were tilted in only the Y-axis of rotation; analysis of rotations about all axes are completed elsewhere [9].

A diagram of a Berkovich tip with an offset angle to the sample being tested is shown in Figure 10. The projected contact area, $A_p$, for an ideal Berkovich tip with a misalignment in the Y-axis by a rotation angle of $\alpha$, is given by

$$A_p = 3.766h^2 \cos\alpha\left(\left(\tan 65.3+\alpha\right)+\tan\left(77.05-\alpha\right)\right)\left(1+\left(\left(\cos\alpha\tan\left(65.3+\alpha\right)-\sin\alpha\right)\sin\alpha\right)\right)$$

Figures 11 and 12 show the percent differences in the projected contact areas and the percent error, respectively, in the corresponding mechanical properties, respectively, over the tilt angle range of ±5°. The results show that the errors are not symmetric — which is expected with a Berkovich tip — and the error in the projected contact area quickly exceeds 10%. Just as in the measurement errors for surface roughness, the hardness results are affected greater by the tilt angle than are the modulus results.

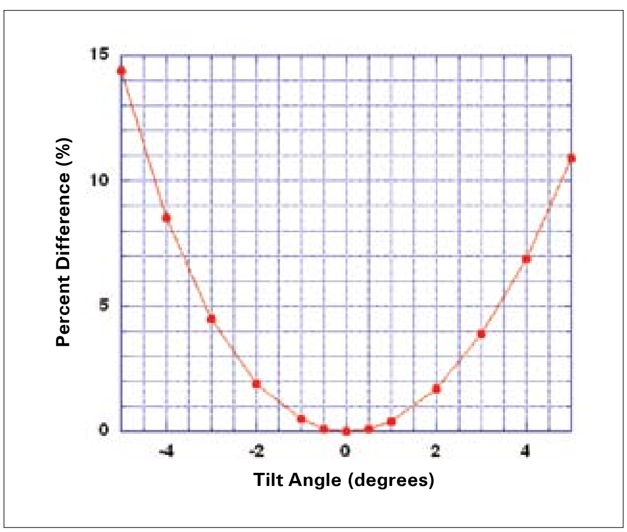


Figure 11. Percent difference in the projected contact area as a function of tilt angle.

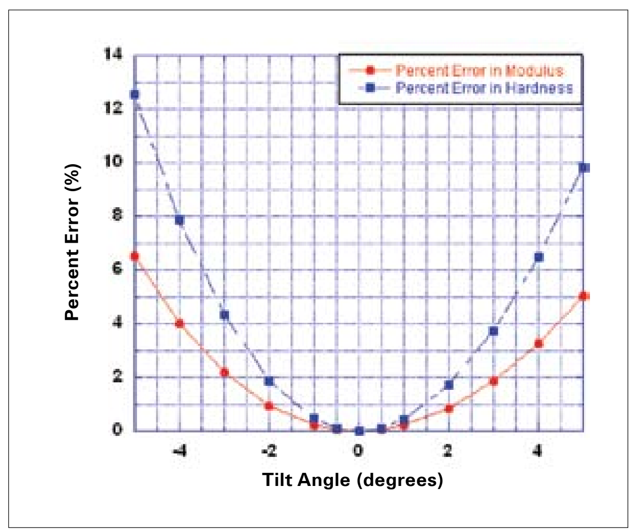


Figure 12. Percent error in mechanical properties based on errors in the calculated lead terms.

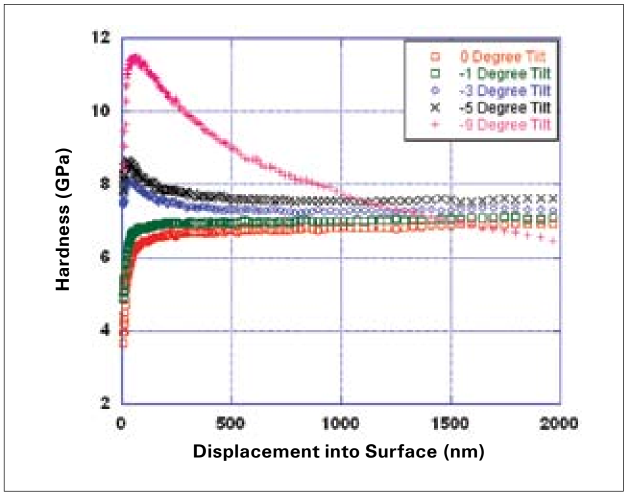


Figure 13. Hardness results as a function of displacement into surface for the tilted samples.

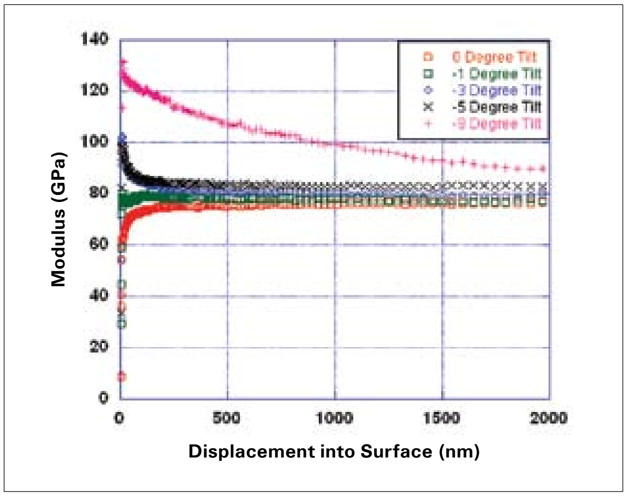


Figure 14. Elastic modulus results as a function of displacement into surface for the tilted samples.

Figures 13 and 14 display the results for the hardness and the elastic modulus, respectively, of the glass slide tested on the five angles. Both the hardness and elastic modulus showed increases in the results of the measured mechanical properties as the offset angle was increased. This was expected since the contact area increases as a result of tilt in the sample, which creates a physical contact that is larger than the instrument is calculating by analyzing the data assuming orthogonal alignment. However, what was unexpected was the amount of error that occurred at the surface for the angles measuring greater than a 1 degree tilt. This error is most likely due to lateral forces caused by the contact of the indenter with a tilted surface. When a non-ideal indenter, an indenter with rounding at the apex, comes into contact with a tilted surface, the edge or the face of the pyramid contacts the surface in manner that causes lateral forces to develop and this exacerbates the tilt angle problem causing larger errors in the calculation of the projected contact area; this is also assumed to be the reason for the errors that were larger than predicted in the measurements of elastic modulus and hardness listed in Table 1. The results from the tilt tests reinforce the notion that the sample surface should be within 1° of orthogonal alignment to axis of the indenter.

| Offset Angle (degrees) | Hardness (GPa) | % Error | Modulus (GPa) | % Error |
|---|---|---|---|---|
| **0** | 6.8 | 0.0 | 75.7 | 0.0 |
| **1** | 7.0 | 2.0 | 78.2 | 3.2 |
| **3** | 7.3 | 6.2 | 80.0 | 5.7 |
| **5** | 7.6 | 10.4 | 83.0 | 9.6 |

Table 1. Errors in the measurements of elastic modulus and hardness at 1000 nm of penetration with respect to sample offset angles.

## Conclusions

The three *Rules of Thumb* for nanoindentation proved to be steadfast rules. The *10% Rule* was the most questionable rule because, often, it is applied very generally to all mechanical properties, as opposed to its original intention as bounds for hardness measurements. In testing the low-k film the *10% Rule* appeared to work very well for both the elastic modulus and hardness. It was observed that soft metals on hard substrates, such as gold films on silicon, can experience pile-up which creates a projected contact area that is greater than predicted by the Oliver-Pharr method. This sample showed a plateau in the hardness results but the hardness was higher than expected due to a combination of pile-up and indentation size effect. The elastic modulus of the gold film was appropriately measured to a penetration depth that was approximately 1% of the film thickness. Tests on the TiN coating showed a plateau in the hardness results but did not show a plateau in the results for elastic modulus. For clear applicability of the *10% Rule*, mechanical properties data should be collected continuously as a function of penetration into the film so that the evolution of mechanical properties can be evaluated for surface and substrate effects.

Results for both the smooth glass slide and the rough steel sample showed that the *5% Rule* is in excellent agreement with acceptable scatter. The measured mechanical properties at the minimum penetration depth, $h_{min}$, given by

$$h_{\min} = \frac{SurfaceRoughness}{0.05}$$

commonly provides results with a covariance less than five percent. However, it was also found that repeatable and reliable measurements could be made at penetration depths that were less than the minimum depth specified by the *5% Rule*; at the shallower penetration depths the data experienced larger covariance in the results.

Results from testing the *1 Degree Rule* for maximum sample tilt were in line with recommendations provided by ISO 14577. The analytical results showed that acceptable errors could withstand up to a 3° tilt; however, in application, sample tilts over 1° showed lateral forces on the indenter tip that exacerbated the tilt angle creating larger errors than predicted. Therefore, sample surfaces should be held within 1° of orthogonal alignment to the indenter.

The Continuous Stiffness Measurement (CSM) option on the Nano Indenter G200 was instrumental in testing the *Rules of Thumb* presented in this article. This option allows observation of the evolution of mechanical properties as the indenter penetrates the surface of the sample. Data from the CSM clearly show influences of substrate and surface effects.

## References


[1] Fischer-Cripps, A. C. Introduction to Contact Mechanics. 2nd ed. New York: Springer Science, 2007.

[2] J.L. Hay and G.M. Pharr. "Instrumented Indentation Testing." *ASM Handbook* 8 (2000).

[3] "Metallic Materials – Instrumented indentation test for hardness and materials parameters." ISO 14577, 2002.

[4] A. Rar, H. Song, and G.M. Pharr. "Assessment of new relation for the elastic compliance of a film-substrate system." *Mater. Res. Soc. Symp.* Proc. 695, pp. 431–438 (2002).

[5] http://www.Goodfellow.com

[6] Tabor, D. The Hardness of Metals. 1951. New York: Oxford University Press, 2000.

[7] W.C Oliver and G.M. Pharr. "Measurement of Hardness and Elastic Modulus by Instrumented indentation: Advances in Understanding and Refinements to Methodology." *Journal of Materials Research* 19 (2004).

[8] J.L. Hay. "Measuring Substrate-independent Modulus of Dielectric Films by Instrumented Indentation." *Journal of Materials Research* 24, (2009).

[9] J.D. Ellis, S.T. Smith, and R.J.Hocken. "Alignment uncertainties in ideal indentation styli." *Precision Engineering* 32, pp. 207–214 (2008).





Printed in USA, April 7, 2010
5990-5700EN


Agilent Technologies