

ADVANCED NANOMECHANICAL TESTING



Effect of surface detection error due to elastic–plastic deformation on nanoindentation measurements of elastic modulus and hardness

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Elastic modulus and hardness are commonly measured using nanoindentation. Calculating these properties from measured force–displacement curves typically requires knowledge of the contact depth of the indenter into the specimen. However, surface detection methods in many nanoindentation experiments can lead to an error in the contact depth measurement and, subsequently, the measured properties. Here, the contributions of elastic and plastic deformations to surface detection errors in nanoindentation experiments are examined through experiments and modeling. The model is used to quantify errors in elastic modulus and hardness measurements due to elastic–plastic deformation during surface detection as a function of the specimen properties, indenter geometry, preload, and contact depth. Nanoindentation measurements on polystyrene, an aluminum alloy, and fused silica specimens with a Berkovich indenter are used to illustrate the effects of surface detection error and are compared to the model. The experiments and model both demonstrate that surface detection error can lead to measurement of apparent depth-dependent properties in homogenous materials.

Introduction

Nanoindentation is a small-scale mechanical characterization technique that is commonly used to measure elastic modulus and hardness of materials. Unlike traditional indentation techniques, nanoindentation typically does not rely on images of the resulting indent after the test to determine the area of the indent; rather, the analysis uses a calibrated area function that quantifies the projected area of the indenter as a function of its contact depth into the specimen. Errors in detecting the surface in nanoindentation measurements can result in incorrect apparent contact depths and thus incorrect areas calculated from the area function. Thus, surface detection errors can lead to errors in modulus and hardness values determined from the nanoindentation data, as the area of the indent is central in the calculations of these quantities. The objective of this paper is to develop a framework for understanding the effect of surface detection errors and quantify the error in hardness and modulus measured via nanoindentation.

The approach developed by Oliver and Pharr [1, 2] is widely used to determine the elastic modulus and hardness from nanoindentation measurements using an indenter with a known area function. The area function is expressed in terms of contact depth into the specimen, thus it is critical to identify the displacement at which the indenter tip first contacts the specimen's surface. There are three common methods used for surface detection in nanoindentation experiments: (1) detection of a preload, (2) monitoring the slope of the loading curve, and (3) use of continuous stiffness measurements. The preload method, which is the focus of the current paper, identifies the surface location as the transducer displacement at which a prescribed preload is exceeded as the tip approaches the specimen. The load threshold is set high enough relative to the measurement noise to be confident that the indenter tip has made contact with the surface, but as low as possible to minimize the amount that the tip of the indenter penetrates the surface; 2 μN is a typical threshold. Since the transducer displacement at which

the preload is reached is defined as zero displacement, any penetration of the tip into the surface due to this preload results in surface detection error.

The effect of surface detection error has been considered previously [3–7], and a quantitative model that considered elastic deformations was developed by Qian et al. [7] for slope-based surface detection systems with a Berkovich indenter, but their analysis can easily be extended to preload-based systems. Their model only considers elastic surface detection errors and is applicable to many materials, but it cannot be used to analyze measurements in which significant plastic deformation occurs during surface detection [8, 9]. While the forces are small in preload detection schemes, the contact areas are also small and thus the stresses can exceed the yield strength of many materials. It will be shown here that this yielding of the material during surface detection can lead to meaningful surface detection errors that must be accounted for in the analysis of certain nanoindentation measurements, particularly measurements at shallow indentation depths and on materials with low hardness.

This paper is structured as follows. First, we briefly review the calculation of hardness and modulus from standard nanoindentation measurements. We then develop mechanics-based models that quantify the error in hardness and modulus measurements resulting from elastic and plastic deformations during preload-based surface detection. Models are presented for pyramidal Berkovich [10] and spherical indenters, which are the two most common indenter geometries. These models are then used to predict errors in hardness and modulus measurements as a function of the material properties of the specimen and test parameters, including the magnitude of the preload and maximum contact depth. Nanoindentation measurements with a Berkovich tip on polystyrene, an aluminum alloy, and fused silica are then used to validate the model.

Background

The Oliver–Pharr Method

The approach developed by Oliver and Pharr [1, 2] for determining elastic modulus and hardness from elastic–plastic nanoindentation measurements relies on the contact depth, h_c . The contact depth is equal to the difference between the maximum displacement into the material, h_m , and the elastic deformation of the material's surface in the region out of contact with the indenter, h_s . This elastic deformation is calculated using the contact stiffness, S , measured as the tangent slope at the peak of the unloading curve, giving the contact depth as

$$h_c = h_m - h_s = h_m - \varepsilon \frac{P_m}{S} \quad (1)$$

where P_m is the load at h_m , and ε is a dimensionless geometric factor that accounts for indenter shape and is approximately

equal to 0.72 for conical indenters, and 0.75 for spherical indenters [11].

The hardness, H , is determined from the peak load and the projected area of the indenter,

$$H = \frac{P_m}{A(h_c)} \quad (2)$$

where $A(h_c)$ is the projected area of the indenter at contact depth h_c . The reduced elastic modulus of the combined indenter and specimen system, E_r , is calculated from the contact stiffness, S , and $A(h_c)$ as

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A(h_c)}} \quad (3)$$

where $\beta = 1.034$ is a dimensionless geometric factor [12], and E_r is defined as $(1/E_r) = ((1 - \nu_i^2)/E_i) + ((1 - \nu^2)/E)$, where E_i and ν_i are the Young's modulus and Poisson's ratio of the indenter, respectively, and E and ν are the properties of the specimen. Calculation of hardness and modulus both require the area of the indent (Eqs. (2) and (3)). Typically, $A(h_c)$ is determined from the calibrated area function of the tip and the contact depth, h_c . Surface detection errors affect the accuracy of h_c and thus affect the area used to calculate hardness and modulus.

Mechanics analysis of surface detection error

Error in measuring reduced modulus and hardness

To assess the effect of surface detection error, we assume there is an error δ_m resulting from penetration of the indenter tip into the specimen during surface detection. Throughout this work, we will refer to values that do not account for this error as “*apparent*”, and values that would be reported if this error is either corrected for or does not exist as “*actual*”. The *actual* maximum displacement, h_m , is the sum of the surface detection error and the *apparent* maximum displacement, h'_m :

$$h_m = h'_m + \delta_m. \quad (4)$$

Throughout this paper a prime (') will be used to denote variables denoting *apparent* quantities. Equation (4) results in a uniform translation of the loading and unloading curves along the displacement axis, thus h_s is unaffected by this error. Given this and Eqs. (1) and (4), the *actual* contact depth, h_c , in terms of the *apparent* contact depth, h'_c , is

$$h_c = h'_c + \delta_m. \quad (5)$$

The *actual* hardness (Eq. (2)) and *actual* reduced modulus (Eq. (3)) in terms of the *apparent* contact depth and the surface detection error are

$$H = \frac{P_m}{A(h'_c + \delta_m)} \quad (6)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A(h'_c + \delta_m)}}. \quad (7)$$

If surface detection error is present and not accounted for, the *apparent* hardness, H' , and *apparent* reduced modulus, E'_r , are

$$H' = \frac{P_m}{A(h'_c)} \quad (8)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A(h'_c)}}. \quad (9)$$

The ratio of the *actual* to the *apparent* properties depends on the area function of the tip, the *apparent* contact depth, and the surface detection error, as shown in Eqs. (10) and (11). To simplify the presentation, $\Phi(h'_c, \delta_m)$, a non-dimensional function of the *apparent* contact depth and the surface detection error, is defined:

$$\frac{H}{H'} = \frac{A(h'_c)}{A(h'_c + \delta_m)} = \Phi(h'_c, \delta_m)^2 \quad (10)$$

$$\frac{E_r}{E'_r} = \sqrt{\frac{A(h'_c)}{A(h'_c + \delta_m)}} = \Phi(h'_c, \delta_m). \quad (11)$$

As δ_m gets larger, or h'_c gets smaller, $\Phi(h'_c, \delta_m)$ departs from unity and the errors in the modulus and hardness measurements become larger. The *actual* material properties can be determined from the *apparent* values and $\Phi(h'_c, \delta_m)$, if the magnitude of the surface detection error is known.

The above analysis is general and can predict the error in modulus and hardness measurements for a given δ_m , regardless of the origin of the surface detection error. δ_m can be can arise from multiple sources, including elastic deformation, plastic deformation, creep, and overshoot of the preload during initial contact. Below, models are presented to estimate δ_m arising from elastic and plastic deformation during surface detection.

Surface detection error due to elastic deformation

If the preload applied during surface detection does not cause yielding, the surface detection error will be due to elastic deformation alone, with $\delta_m = \delta_e$, where δ_e is the elastic deformation of the contact at the preload, P_0 . From classical Hertz contact mechanics, δ_e for a spherical indenter in contact with an elastic half-space is

$$\delta_{e-Sph} = \left(\frac{3P_0}{4\sqrt{RE_r}} \right)^{2/3} \quad (12a)$$

where R is the indenter radius. The elastic contact mechanics of conical indenters was considered by Bousinesq and later Sneddon [13], and applied to Berkovich indenters by Bilodeau [14],

who gives the elastic deformation of a Berkovich indenter in contact with an elastic half-space as

$$\delta_{e-Berk} = \left(\frac{2P_0}{k \tan(\alpha) E_r} \right)^{1/2} \quad (12b)$$

where $k \approx 1.7773$ and α is the cone half-angle ($\alpha \approx 70.3$ degrees for typical Berkovich indenters).

To facilitate the application of the above to experiments in which surface detection error is present, we rewrite the surface detection error in terms of the *apparent* reduced modulus, E'_r , and $\Phi(h'_c, \delta_m)$, which is a function of the *apparent* contact depth, h'_c . Using Eq. (11), Eqs. (12a) and (12b) are rewritten as

$$\delta_{e-Sph} = \left(\frac{3P_0}{4\sqrt{RE'_r}} \Phi(h'_c, \delta_{e-Sph})^{-1} \right)^{2/3} \quad (13a)$$

$$\delta_{e-Berk} = \left(\frac{2P_0}{k \tan(\alpha) E'_r} \Phi(h'_c, \delta_{e-Berk})^{-1} \right)^{1/2}. \quad (13b)$$

Depending on the form of $\Phi(h'_c, \delta_m)$, these equations can be solved either analytically or numerically to determine δ_e .

Surface detection error due to plastic deformation

If the preload induces sufficient plastic deformation in the specimen, the surface detection error can be predicted using the Oliver and Pharr framework. From Eq. (1), the surface detection error due to plastic deformation is

$$\delta_m = \delta_c + \varepsilon \frac{P_0}{S} \quad (14)$$

where δ_c is analogous to h_c , and S is the contact stiffness at depth δ_c . S cannot typically be determined in the surface detection process, but can instead be found using Eq. (3), giving the surface detection error as

$$\delta_m = \delta_c + \left(\frac{\epsilon \sqrt{\pi}}{2\beta} \right) \frac{P_0}{\sqrt{A(\delta_c) E_r}}. \quad (15a)$$

The surface detection error contact depth, δ_c , is related to the preload and material hardness by Eq. (2) as

$$A(\delta_c) = \frac{P_0}{H}. \quad (15b)$$

Equations (15a) and (15b) are in terms of the *actual* material properties. However, it is often more convenient experimentally to have these expressions in terms of the *apparent* properties, thus, using Eqs. (10) and (11), these expressions are rewritten as

$$\delta_m = \delta_c + \left(\frac{\epsilon \sqrt{\pi}}{2\beta} \right) \frac{P_0}{\sqrt{A(\delta_c) E'_r}} \Phi(h'_c, \delta_m)^{-1} \quad (16a)$$

$$A(\delta_c) = \frac{P_0}{H'} \Phi(h'_c, \delta_m)^{-2}. \quad (16b)$$

In Eqs. (16a) and (16b), E'_r and H' are calculated at the *apparent* contact depth h'_c . These two independent equations form a system written entirely in terms of measured *apparent* values as well as δ_c and δ_m , and they can therefore be solved either analytically or numerically for δ_c and δ_m , of which the latter is the surface detection error.

Limits of the elastic and plastic models

The elastic and elastic–plastic models for surface detection error presented in this work are expected to be accurate under the constraints for fully elastic or fully plastic indents, respectively. While the mechanical response of materials beyond yield is innately system-specific, with the behavior during indentation depending on a specimen's particular constitutive relationship, as well as the geometry of the indenter and the surface interactions between the specimen and the indenter (i.e. friction) [15], the conditions of the onset of yielding and fully plastic indentation have been extensively investigated in the literature [16–18]. These models assume frictionless indentation and an elastic-perfectly-plastic specimen but provide reasonable estimates of the transition from elastic to plastic deformation for many materials and are useful for understanding the limits of the elastic and elastic–plastic surface detection error models.

The equations for elastic surface detection error ((12a)–(13b)) are only valid if there is no inelastic (plastic) deformation. The onset of yielding during indentation is predicted for many materials with a well-defined yield strength, Y , using the Tresca criterion. Other yield conditions such as the Von Mises or reduced stress conditions give similar predictions but are less convenient. For indentation with a spherical indenter, the Hertz solution and the Tresca criterion predicts yielding will occur at an applied load, P_Y , of [19]

$$P_Y = \frac{1}{6} \left(\frac{R}{E_r} \right)^2 (1.6\pi Y)^3. \quad (17)$$

For indentation with a conical tip, which is often used for the analysis of a Berkovich indenter [20], the condition [15] for yielding is

$$\cot(\alpha) \geq \frac{Y}{E_r} \quad (18)$$

Due to the stress singularity at the tip of a perfect cone and the cone's self-similarity, the condition for yielding is independent of the applied load.

The yield strength, Y , is related to the hardness measured via nanoindentation by [21]

$$Y = \frac{H}{\kappa} \quad (19)$$

where κ is a constant that depends on the mechanical properties of the material and the indenter geometry. For many materials and indenter geometries, the value of κ is between 2 and 3 [22–24], but in some cases it may be outside these bounds. For this work, κ is taken as 3, although the effect of a lower κ value is also highlighted and this model can readily be adapted to different values for κ . In this model, κ is only used to determine the limit of the elastic surface detection error prediction.

With the assumption of $\kappa = 3$, the conditions under which the plastic strains become dominant in the indent occurs when the mean stress plateaus at $\sim 3Y$ [25], which is referred to as a fully plastic indent. For a spherical indenter [26], the applied load, P_{FP} , for a fully plastic indent is [15]

$$P_{FP} \approx 400P_Y. \quad (20)$$

For a conical indenter, the criterion for a fully plastic indent is [15]

$$\cot(\alpha) \approx 30 \frac{Y}{E_r}. \quad (21)$$

The elastic model and elastic–plastic models for surface detection error derived in this work are expected to be accurate under the constraints for fully elastic or fully plastic indents, respectively. Since the exact behavior when there is local plasticity (i.e. the deformation at loads just beyond the onset of yield), cannot be formulated in a general way, we use the elastic–plastic model for all cases in which yielding is predicted. This is a simplification and the elastic–plastic model will initially over-predict the error in the region just beyond the limits of the elastic model. However, we expect the elastic–plastic analysis will be quantitatively accurate for most cases beyond yield, since the plastic zone typically grows under an indenter tip very quickly after yielding, even if the conditions for a fully plastic indent are not achieved.

Modeling results

Numerical solutions to the model are presented for three area functions: an ideal Berkovich indenter, a blunted Berkovich indenter, and a spherical indenter. Results are given for ideal area functions for generality and to provide estimates of the error. In the case of the ideal Berkovich area function, the assumption of a perfectly sharp indenter will result in an overestimate of the error relative to a real indenter with a rounded tip. The impact of tip rounding or blunting is evident in the results for the blunted Berkovich indenter below.

An ideal berkovich indenter

For the Berkovich indenter, the ideal area function is

$$A_{Berk}(h_c) = 24.5h_c^2 \quad (22)$$

The ratios of the *actual* reduced modulus and hardness to the *apparent* properties are given in Fig. 1 as a function of the *apparent* hardness and reduced modulus. A smaller ratio in the plots indicates a larger error. The results are given for a preload of $P_0 = 2 \mu\text{N}$, and an *apparent* contact depth of $h_c^* = 100 \text{ nm}$. In the experiments for this work, three materials are tested: polystyrene (PS), an aluminum alloy (Al), and fused silica (FS). For reference, the PS and Al are shown on the maps in Fig. 1; the fused silica is not shown as its properties place it outside the bounds of the map.

When a material has sufficiently high hardness relative to its modulus (i.e., the lower right regions of the maps in Fig. 1), the error is due to elastic deformation and is independent of the hardness. For materials with lower relative hardness, the error is dominated by the plastic deformation and the hardness is the primary factor in determining the error.

The ideal Berkovich area function assumes an indenter with a perfectly sharp point, whereas any real indenter will have some blunting. This means that the modeling results presented here will overestimate the error when the error is small (i.e., the upper right regions of the maps in Fig. 1). These results are shown for an *apparent* contact depth of 100 nm, which is relatively shallow, but shallow indentation depths are frequently used to avoid substrate effects when measuring properties on thin films using nanoindentation.

The model uses a yield criterion defined by Eqs. (18) and (19), with $\kappa = 3$; however, the condition for $\kappa = 2$ is also shown in Fig. 1. Between these boundaries, the elastic model is a lower bound on the error and the plastic model acts as an upper bound. Additionally, the predicted error at the yield

criterion boundary is not smooth and continuous. This arises from the assumption that the indent is fully plastic in the elastic-plastic model, which is not the case immediately after the onset of yield. This leads to a slight overprediction in the error relative to the elastic result, but this overprediction is typically less than 10% greater than the elastic prediction (as can be seen in the weakness of the discontinuity in Fig. 1).

The results in Fig. 1 are shown for one set of experimental conditions; however, the total amount of error depends nonlinearly on the contact depth and the applied preload. The effect of the error decreases as the *apparent* contact depth increases. Results as a function of *apparent* contact depth for four example material property combinations are given in Fig. 2. In general, indents performed at low loads, and hence low contact depths, incur more significant errors since the relative size of the surface detection error to the contact depth becomes larger.

The surface detection error increases with the magnitude of the preload, thus the effect of surface detection error on modulus and hardness measurements can be reduced by decreasing the preload, as shown in Fig. 3. While nanoindenter force transducers with lower noise floors may allow for preloads less than $2 \mu\text{N}$, this value is frequently used as a minimum for the testing of a wide range of materials. Small variations in the preload around $2 \mu\text{N}$ will not cause large variations in the amount of error for most materials, relative to the strong dependence on *apparent* contact depth (Fig. 2). Furthermore, even if the preload is decreased, there can still be substantial surface detection errors for materials with lower values of modulus and hardness, as seen in Fig. 3.

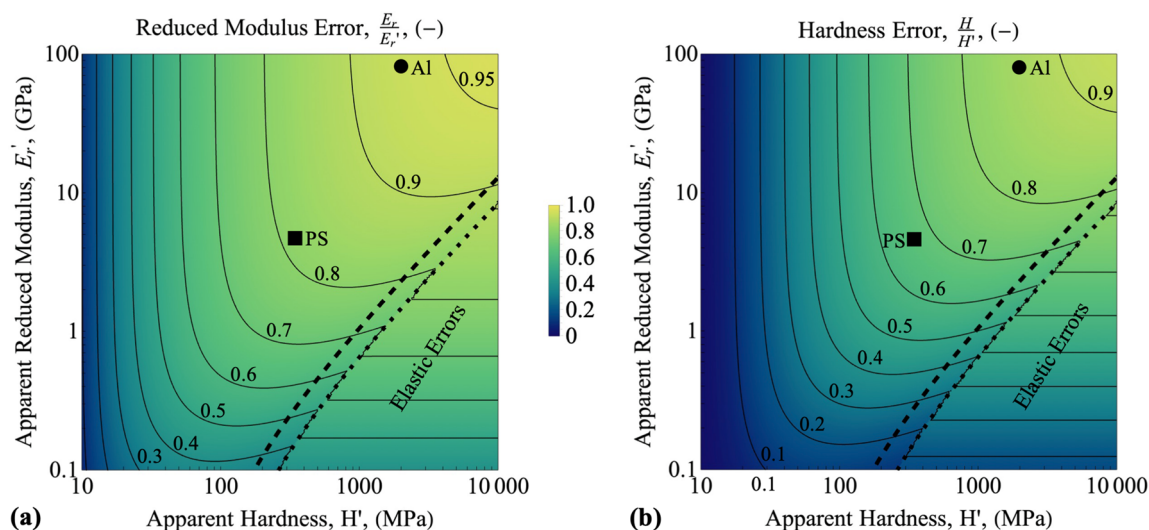


Figure 1: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* values as a function of the *apparent* properties for an ideal Berkovich indenter. Results shown are for a preload $P_0 = 2 \mu\text{N}$, and an *apparent* contact depth of $h_c^* = 100 \text{ nm}$. The dotted lines indicate the yielding condition with $\kappa = 3$, and dashed lines indicate the yield condition with $\kappa = 2$. Square markers indicate results for polystyrene (PS), and circle markers indicate results for an aluminum alloy (Al), based on the material properties given in Table 1.

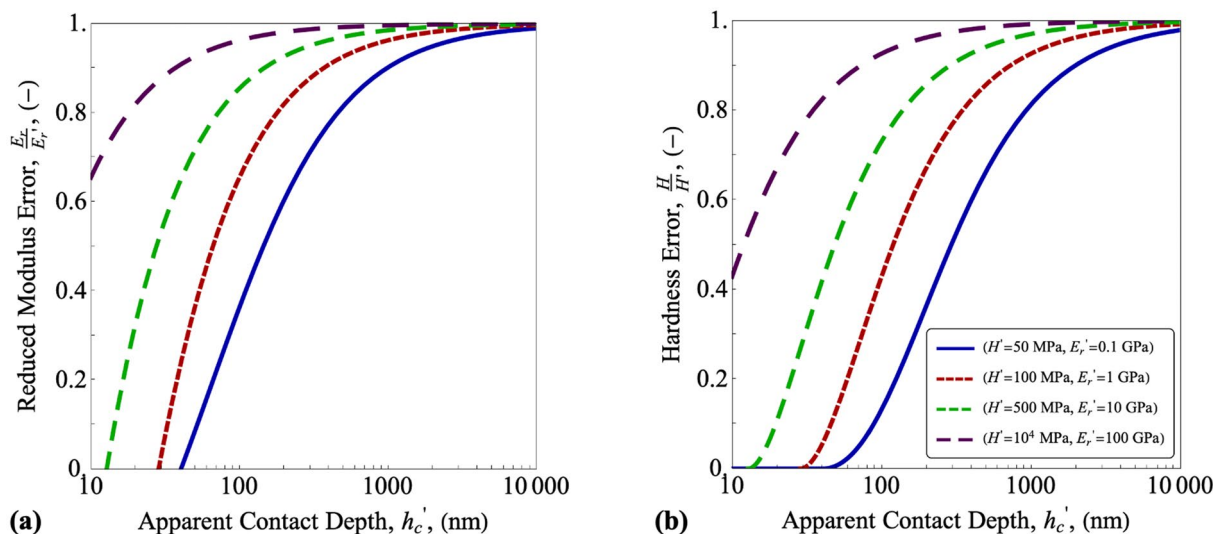


Figure 2: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of the *apparent* contact depth is given for four example combinations of *apparent* reduced modulus and hardness, for an ideal Berkovich indenter area function and a preload $P_0 = 2 \mu\text{N}$.

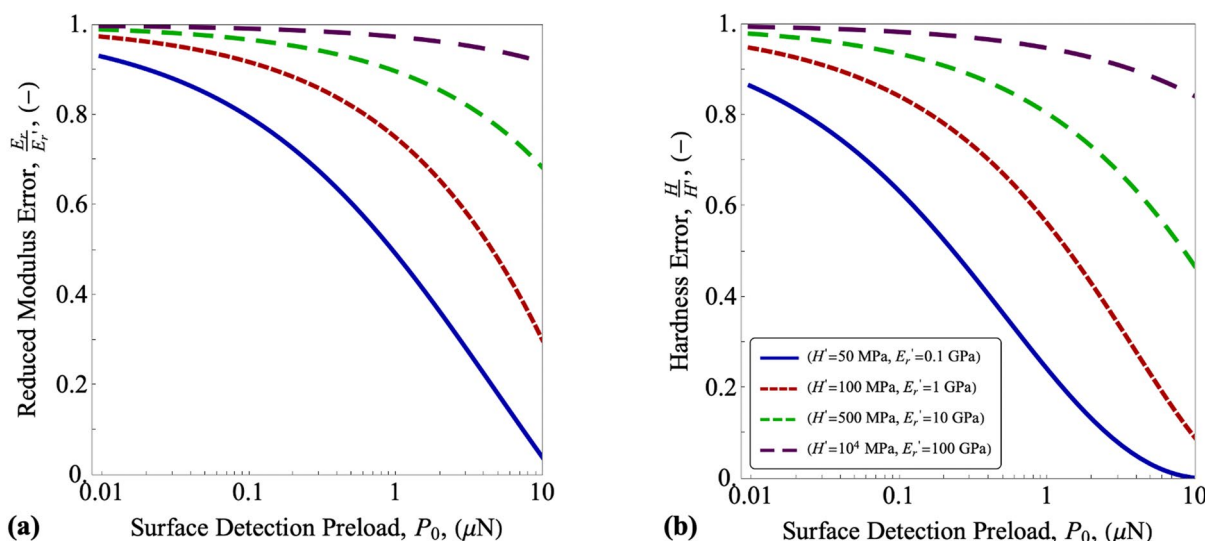


Figure 3: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of the surface detection preload is given for four example combinations of *apparent* reduced modulus and hardness, for an *apparent* contact depth $h_c^* = 100 \text{ nm}$, with an ideal Berkovich indenter.

A blunted Berkovich indenter

Since all real pyramidal indenters will have some blunting at the tip, modeling results for an idealized blunted Berkovich indenter in which the tip has a finite radius of curvature are considered here. The area function has the form:

$$A_{\text{Blunted}}(h_c) = \begin{cases} \pi (2Rh_c - h_c^2), & h_c < D \\ 24.5(B + h_c)^2, & h_c \geq D \end{cases} \quad (23)$$

where R is the radius of curvature of the blunted end of the indenter, $D \approx 0.106R$, and $B \approx 0.054R$, which ensures

continuity and smoothness of the area function. The ratios of the *actual* reduced modulus and hardness to the *apparent* properties are given in Fig. 4 for a preload of $P_0 = 2 \mu\text{N}$, a measured contact depth of $h_c^* = 100 \text{ nm}$, and a tip radius of $R = 150 \text{ nm}$, which is a typical value for a relatively new Berkovich tip.

As a result of the blunted tip, at the common preload value of $2 \mu\text{N}$, the surface detection indent is elastic for a larger range of material properties than the ideal Berkovich prediction, assuming $\kappa = 3$ or $\kappa = 2$ (Fig. 4). However, due to the constraints of Hertzian elastic models, which require the deformation to be small ($< 10\%$), relative to the radius of the tip, the elastic error

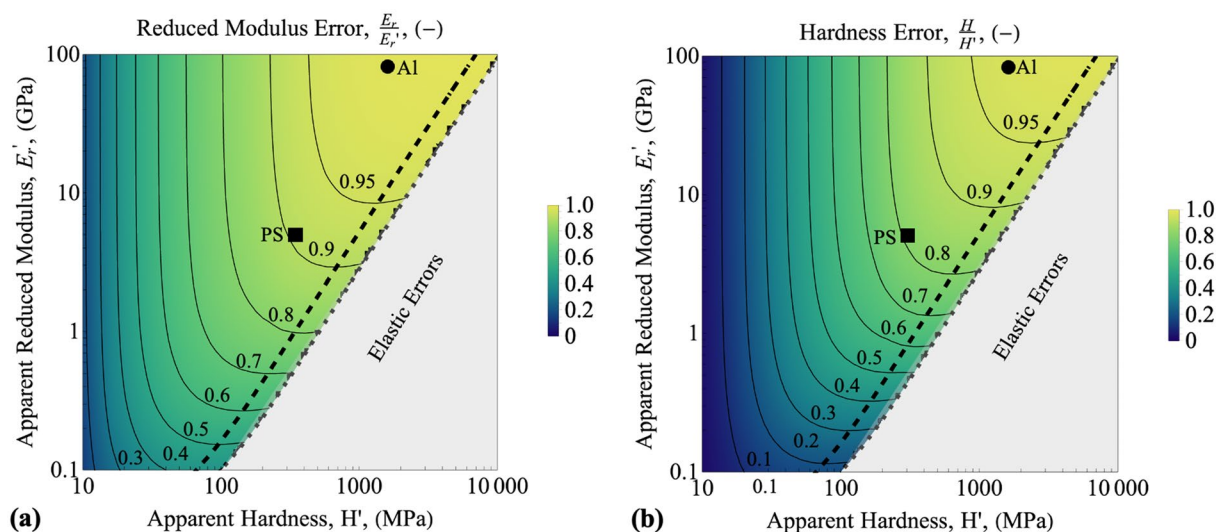


Figure 4: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* values as a function of the *apparent* properties for a blunted Berkovich indenter, with a tip radius $R = 150$ nm. Results shown are for a preload $P_0 = 2$ μ N, and an *apparent* contact depth of $h_c' = 100$ nm. The dotted lines indicate the yielding condition with $\kappa = 3$, and dashed lines indicate the yield condition with $\kappa = 2$. No results for elastic deformation are presented. Square markers indicate results for polystyrene (PS), and circle markers indicate results for an aluminum alloy (Al), based on the material properties given in Table 1.

is not easily calculated in this range of material properties, since some material combinations may violate this condition. Thus, the elastic surface detection error results are not included for these maps. As can be seen in the ideal Berkovich results (Fig. 1), the errors due to elastic deformation will always be smaller than those resulting from plastic deformation, for a given *apparent* reduced modulus.

In general, the plastic errors incurred by the blunted Berkovich tip (Fig. 4) are smaller than those predicted by the ideal Berkovich (Fig. 1). The decrease in error is most significant when the errors are small, with the aluminum alloy incurring almost no error for the blunted Berkovich model ($\sim 2\%$ in hardness and $\sim 1\%$ in reduced modulus) compared to the more significant errors predicted by the ideal model ($> 5\%$ error in modulus and $> 10\%$ error in hardness). Given a typical blunting of 150 nm, many materials can still incur measurable or significant errors, as evidenced by the result for polystyrene shown in Fig. 4, which still sustains a predicted $\sim 8\%$ error in modulus and $\sim 15\%$ error in hardness.

As with the ideal Berkovich results (Figs. 2 and 3), the amount of error will increase as the *apparent* contact depth decreases, or if a higher preload is used. The error will also depend on the degree of blunting (i.e. the radius of the spherical portion of the tip). Due to the limited span of radii for typical Berkovich indenters, ranging from ~ 50 nm to a few hundred nanometers, the variance in the amount of error due to the degree of tip blunting is small, relative to other effects such as contact depth. Modeling results showing how the error depends on the radius of the blunted end of the indenter are given in the Supplemental Information (Fig. S1).

A spherical indenter

Spherical indenters typically have much larger radii, on the order of 1–10 μ m. For a spherical indenter of radius R , the ideal area function is

$$A_{\text{sph}}(h_c) = \pi (2Rh_c - h_c^2) \quad (24)$$

In general, due to its flatter profile, which causes the contact area to rapidly increase with contact depth, measurements with a spherical indenter have smaller surface detection errors than measurements with an ideal or real Berkovich indenter. As shown in Fig. 5, compared to the Berkovich (Fig. 1) and blunted Berkovich results (Fig. 4), the errors for a spherical indenter of $R = 1$ μ m are much smaller. Moreover, at the preload value of 2 μ N, the surface detection indent is elastic for an even larger set of materials, assuming $\kappa = 3$ or $\kappa = 2$ (Fig. 5). The transition between the models for elastic and elastic–plastic error is smoother in the spherical analysis compared to the ideal Berkovich. This is because the effect of plasticity is initially much smaller under a spherical indenter compared to a perfectly sharp tip.

For less stiff materials, even the elastic deformation under a large spherical indenter can cause measurable amounts of error in the predicted reduced modulus and hardness if the contact depth is not sufficiently deep. Similarly to the result for an ideal Berkovich, the error increases as the *apparent* contact depth decreases or if a high preload is used. Modeling results that quantify how the error depends on the *apparent* contact depth and applied preload for a spherical indenter are given in Figs. S2 and S3, respectively, in the Supplemental Information.

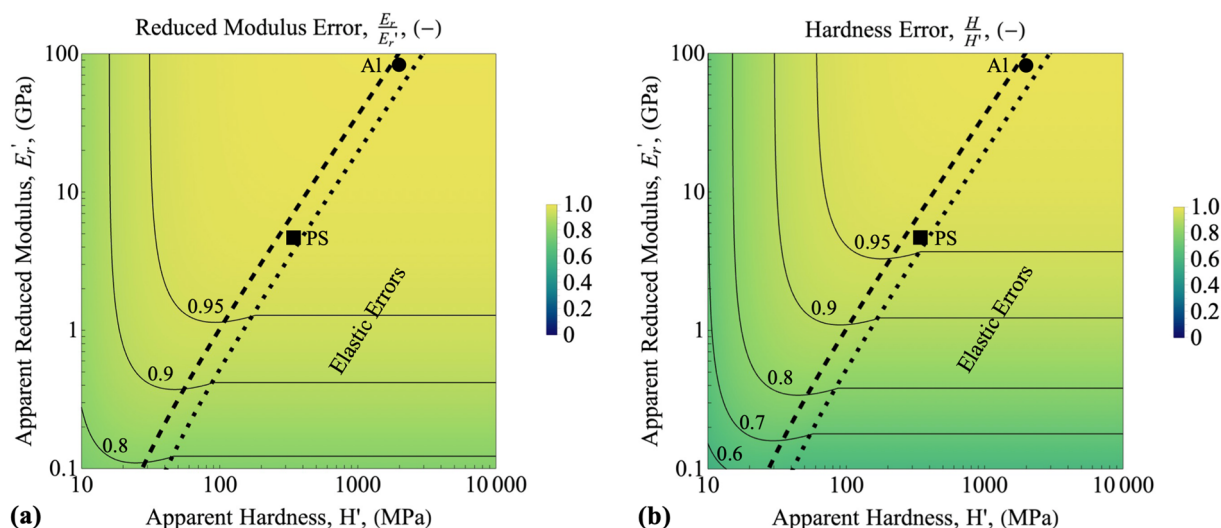


Figure 5: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of the *apparent* properties are given for an ideal spherical area function. Results shown are for a preload $P_0 = 2 \mu\text{N}$, an *apparent* contact depth of $h_c^* = 100 \text{ nm}$, and indenter radius $R = 1 \mu\text{m}$. Dotted lines indicate the yielding condition with $\kappa = 3$, and dashed lines indicates the yield condition with $\kappa = 2$. Square markers indicate results for polystyrene (PS), and circle markers indicate results for an aluminum alloy (Al), based on the material properties given in Table 1

Experimental results

The effect of surface detection error on nanoindentation measurements using a Berkovich indenter was investigated for three common materials: polystyrene (PS), an aluminum alloy (Al), and fused silica (FS). These materials were chosen as they have a range of mechanical properties and thus are expected to have a range of surface detection errors, with polystyrene expected to have the largest error and fused silica expected to have the least amount of error. For each material, two sets of indents were performed to varying contact depths: (1) One set used a standard preload detection scheme in the measurements and did not account for surface detection errors, thus these measurements yield the “*apparent*” properties; (2) the second set used a lift-off and offset technique that allowed surface detection errors to be avoided and thus yield the “*actual*” properties. The experimental methods for both types of measurements are detailed in the Methodology section.

The experimental results for PS are shown in Fig. 6 for three different preloads and a range of contact depths. In the *apparent* measurements (solid circles), the modulus and hardness depend on *apparent* contact depth with higher values at smaller contact depths. This trend becomes stronger with higher preloads. However, in the range of preloads explored here, which represent the limits of typical nanoindentation equipment, the preload only has a small effect, relative to the effect of contact depth.

The *apparent* polystyrene data contrasts strongly with the *actual* data (open triangles) that were measured using the lift-off and offset technique, as described in the methodology section below. The modulus and hardness in these *actual* measurements are largely constant with depth. Table 1 lists

the average and standard deviation of the *actual* modulus and hardness values measured over all depths using the lift-off and offset technique. The small standard deviations (2–4% of the mean values) in Table 1 and the results in Fig. 6 clearly show that the *actual* properties do not vary with depth. A similar lack of depth dependence was observed for the Al and FS measurements and the *actual* modulus and hardness values for these measurements are also given in Table 1 (with standard deviations of 1–6% of the mean values).

To demonstrate the use of the framework presented in this paper for understanding and assessing nanoindentation data, we use the model to predict the surface detection error from the *apparent* measurements. Using Eqs. (16a) and (16b) with the indenter’s calibrated area function, the surface detection error was calculated from the *apparent* results at each contact depth on all specimens. The average surface detection error (across all contact depths) determined through this calculation for each specimen and preload is summarized in Table 2. Such a calculation could be done on existing nanoindentation datasets to understand the magnitude of surface detection error due to elastic–plastic deformation, if this was not previously accounted for.

The surface detection error was also calculated from the *actual* experimental modulus and hardness values, using Eqs. (15a) and (15b), with the result also summarized in Table 2. The surface detection error predicted from the *actual* and *apparent* properties are in reasonable agreement with one another and the differences are due to the *apparent* prediction incurring the effect of any additional errors in surface detection not accounted for in the preload, as discussed below.

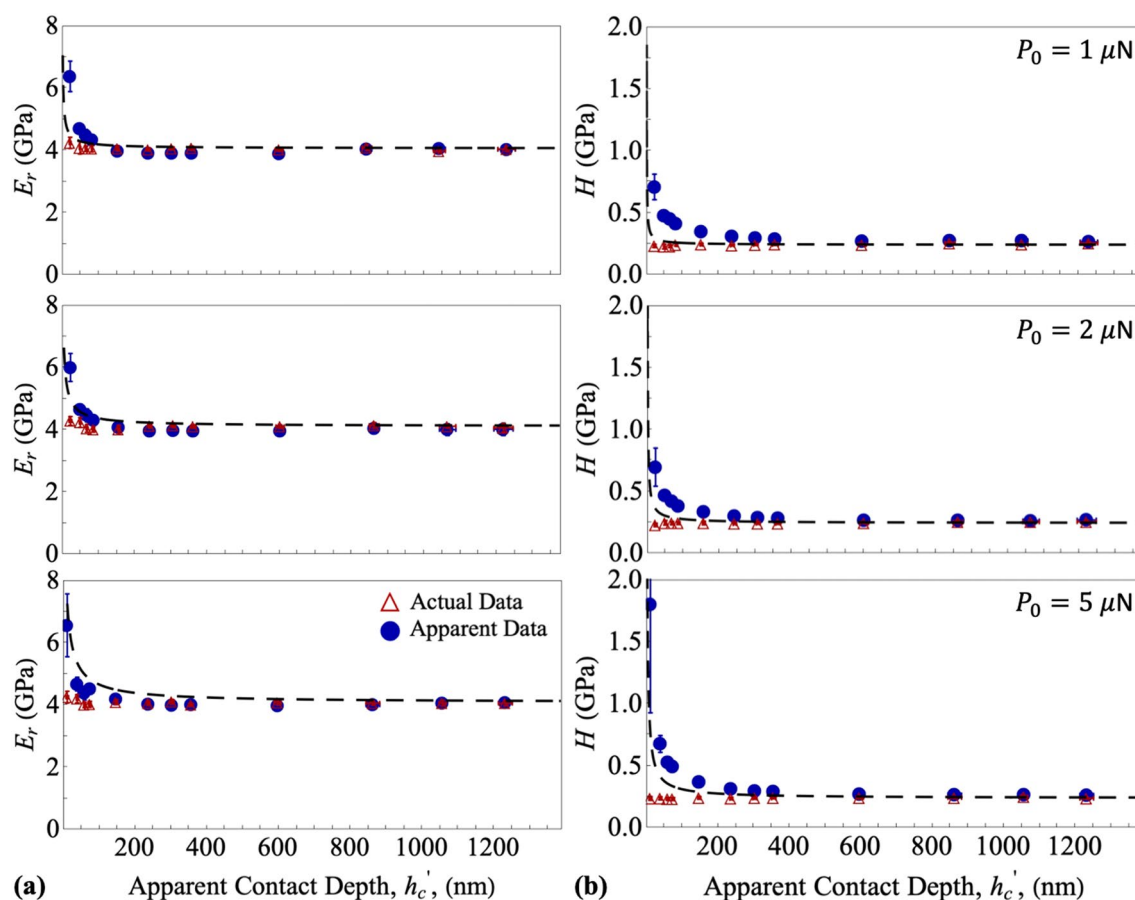


Figure 6: Experimental results of (a) reduced modulus and (b) hardness of polystyrene as a function of the *apparent* contact depth for three surface detection preloads, P_0 . The dashed line indicates the model's prediction of the *apparent* data given the average *actual* data and the preload.

TABLE 1: *Actual* reduced modulus and hardness results (average $\pm$ std. dev.) for each specimen measured using the lift-off and offset technique, and the range of contact depths over which they were measured, rounded to the nearest 5 nm.

Specimen	Reduced Modulus, E_r (GPa)	Hardness, H (GPa)	Range of contact depths (nm)
Polystyrene, $P_0 = 1 \mu\text{N}$	4.1 ± 0.1	0.242 ± 0.009	50–1250
Polystyrene, $P_0 = 2 \mu\text{N}$	4.1 ± 0.1	0.246 ± 0.007	50–1250
Polystyrene, $P_0 = 5 \mu\text{N}$	4.1 ± 0.1	0.239 ± 0.005	50–1260
Aluminum, $P_0 = 2 \mu\text{N}$	78 ± 1	1.7 ± 0.1	25–490
Fused silica, $P_0 = 2 \mu\text{N}$	64 ± 2	9.2 ± 0.1	30–130

The surface detection error predictions due to elastic–plastic deformation were validated through an analysis of the loading curve of the *actual* results. The displacement into the specimen at a force equal to the preload was measured from the *actual* loading curves and compared to the surface detection error predictions. The results are summarized in Table 2. For polystyrene and fused silica, the measurements agree within error of the result calculated from the *apparent* material properties. For the aluminum alloy, the loading curve measurement suggests the

model is underpredicting the amount of deformation by a few nanometers. This is likely due to larger surface roughness on the aluminum specimen, which will make the surface effectively softer than the bulk material. In polystyrene, it is notable that the deformation due to the preload of $2 \mu\text{N}$ is about 7–9 nm, showing that meaningful errors can occur even in materials that are not typically thought of as being soft.

To understand the effect of these elastic–plastic surface detection errors on the measured material properties, Eqs. (10) and

TABLE 2: Surface detection error predictions from the *actual* and *apparent* properties, measured depth of *actual* loading curve at the preload, and the necessary error to match *apparent* and *actual* hardness values (average $\pm$ std. dev.).

Specimen	δ_m calculated from <i>apparent</i> E_r and H measurements (nm)	δ_m calculated from <i>actual</i> E_r and H measurements (nm)	Displacement measured from <i>actual</i> loading curve at preload force (nm)	Calculated total error from hardness model to match <i>apparent</i> and <i>actual</i> results (nm)
PS 1 μ N	5.0 ± 0.7	3.6 ± 0.3	6 ± 1	30 ± 3
PS 2 μ N	9 ± 1	7.2 ± 0.4	8 ± 1	26 ± 2
PS 5 μ N	18 ± 4	16.4 ± 0.9	19 ± 1	39 ± 2
Aluminum 2 μ N	1.1 ± 0.1	0.83 ± 0.05	4 ± 1	8 ± 2
Fused silica 2 μ N	1.4 ± 0.2	0.24 ± 0.03	2 ± 1	4 ± 2

(11) were used to calculate the predicted *apparent* properties from the *actual* material properties (Table 1) and the surface detection error, δ_m , calculated from *actual* E_r and H measurements (Table 2). This result is plotted in Fig. 6 (dashed line). This prediction follows the same trend as the measured *apparent* properties, but underpredicts the amount of error present. This suggests other surface detection errors exist in addition to the elastic-plastic deformation induced by the programmed preload. The most likely source of these errors is additional indenter creep into the specimen during the settle time and drift correction measurement, which occur immediately after surface detection. The indenter can also overshoot the preload during surface detection if the software has any delay in stopping the approach after the preload is achieved, which would also accumulate additional errors.

While investigating the contribution of creep deformation to surface detection is outside the scope of this work, the framework and experimental data provided here may be used to determine the total surface detection error resulting from all sources. Using Eqs. (10) and (11), the *actual* and *apparent* data sets can be used to predict the total surface detection error in the depth measurements, which will be a combination of the elastic/plastic deformation due to the preload, and any additional errors. For all tested *apparent* contact depths on each specimen, Eqs. (10) and (11) were used to calculate the necessary amount of error, δ_m , to bring the *apparent* values in agreement with the

actual measurements. Table 2 lists this total amount of surface detection error, averaged over all contact depths. The calculated error was relatively insensitive to contact depth as reflected by low standard deviations for these values (Table 2). This result is further illustrated in Fig. 7, where the total average error is used to correct the *apparent* hardness for each specimen during indentation with a 2 μ N preload. It is clear that the corrected *apparent* hardness (open circles) matches closely with the *actual* hardness over all contact depths. Hardness measurements are shown in Fig. 7 as the effect of surface detection error is more pronounced than for modulus (compare Eqs. (10) and (11)).

Discussion

The *apparent* measurements, when corrected for surface detection errors (Fig. 7), are consistent with the *actual* properties and independent of contact depth. These results demonstrate that, due to the depth-dependent effects of surface detection error, if it is not accounted for it can lead to apparent depth-dependent material properties or apparent size effects. This does not preclude the possibility of size effects existing in nanoindentation, but rather demonstrates that surface detection errors must be accounted for if size effects are also to be investigated. This point was previously noted by Qian et al. [7], who investigated the significant impact of elastic surface detection errors. The work

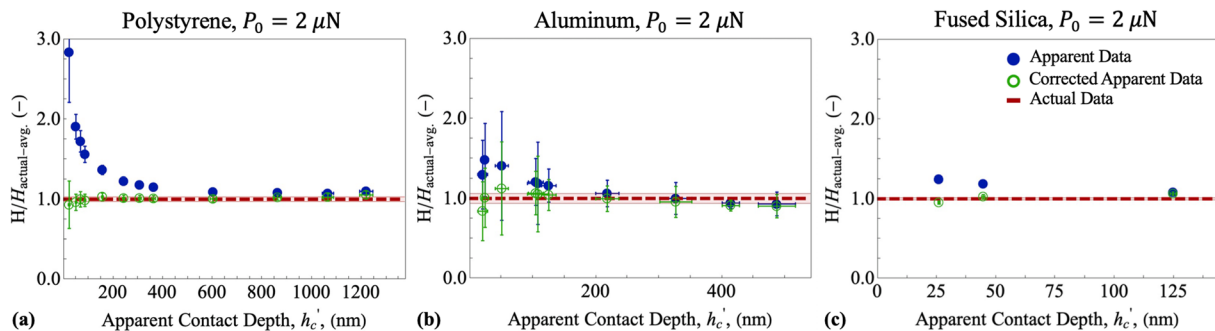


Figure 7: The *apparent* hardness results, the corrected *apparent* hardness results using the average total error, and the average $\pm$ std. dev. of the *actual* hardness results, of (a) polystyrene, (b) aluminum alloy, and (c) fused silica. All results are normalized by the average *actual* hardness of each specimen.

presented here builds on this previous work and shows that plastic deformation can contribute to surface detection errors even when the specimen does not have a low elastic modulus. As seen in Figs. 1 and 3, both the elastic modulus and the hardness affect the error incurred during preload-based surface detection.

The model quantifies the effect of the preload magnitude and shows that surface detection error can be decreased by decreasing the preload (Fig. 3). However, the model and the experimental results for PS (Fig. 6) also show that adjusting the preload only has a small (but measurable) effect on the surface detection error when the preload range is constrained by realistic values for currently available transducers. The model and experimental results show that the contact depth is the key parameter that can be altered to reduce the effect of surface detection errors by performing deeper indents. For polystyrene, at shallow contact depths ($< \sim 600$ nm), the *apparent* hardness and modulus values are higher than the *actual* values due to surface detection error (Fig. 6). For contact depths $> \sim 600$ nm the *apparent* and *actual* values agree within error and no depth dependence is observed in the *apparent* data (Fig. 6). Similar trends are observed for the Al and FS specimens (Fig. 7), but the errors are smaller because of the higher modulus and hardness of these materials. From these experimental results and the modeling results (Fig. 2), it is clear that a simple way to minimize the effect of surface detection errors is to increase the contact depth by performing deeper indents. Increasing the contact depth is straightforward when testing bulk samples but can be challenging in testing thin films where maximum contact depths are limited to a fraction of the film thickness to avoid substrate effects [27].

As shown by the *actual* results in Figs. 6 and 7, the effect of surface detection errors can also be eliminated through experimental practices like the lift-off and offset technique employed here. Experimental techniques such as this are particularly useful when investigating depth-dependent properties and can help reveal if a material has properties that actually vary with depth, or if surface detection error effects are creating the appearance of depth-dependence. In this work, the lift-off and offset technique demonstrated that none of the three materials investigated here (PS, FS, and Al) had properties with a significant depth-dependence.

Two tools are presented here, (1) a method for determining the effect of surface detection errors on the measurements of hardness and modulus (Eqs. (10) and (11)), if the surface detection error is known, and (2) a model for predicting the contributions to the surface detection error from elastic and plastic deformations arising from a surface detection preload. As seen in the data presented here, there can be additional errors in the surface detection due to creep or the precision of the indenter and these are not accounted for in this model. Regardless, the model provides a lower bound on the surface detection error that may be present.

Conclusions

A framework to understand surface detection error due to elastic-plastic deformation in a preload-based surface detection system is presented and applied to examine potential errors in nanoindentation measurements of elastic modulus and hardness. The results show that, in some cases, significant errors due to surface detection error can occur, especially if indents are performed at shallow depths. For a given measured contact depth and preload, the surface detection error will be larger for materials with lower elastic modulus and hardness properties; however, even materials that are often considered to be relatively stiff and hard may incur measurable amounts of error. The results here also show that, due to the depth-dependent nature of the effect of surface detection error, it is possible for a material to appear to exhibit higher reduced modulus and hardness at shallower indents due to unaccounted-for surface detection error.

Methodology

Experimental setup

All experiments in this work were performed using a Hysitron TI-950 nanoindenter fitted with a diamond Berkovich indenter. The system uses a preload-based surface detection scheme. The area function of the indenter and details of how the area function was calibrated are given in the Supplemental Information.

Three specimens were tested, a polystyrene sample cut from the base of a standard petri dish and measured with a caliper to be 654 ± 1 μm thick, a bulk aluminum alloy, and a fused silica specimen. All specimens were mounted on steel pucks and magnetically attached to the nanoindenter stage.

A set of indents were performed on each specimen, utilizing the preload for surface detection, to measure the *apparent* properties. On the aluminum and polystyrene specimens, a set of 40 indents were performed with a 15 μm spacing at each prescribed peak load to ensure neighboring indents did not interfere with each other. Due to the homogeneity of fused silica, only 20 indents were necessary to collect at each prescribed peak load, with a 5 μm spacing to ensure the indents did not interfere with each other. The surface detection error is not accounted for when running these experiments.

The indents were performed under load control with the polystyrene specimen using a 5-s loading time, 5-s hold time, and 1-s unload time, which has been shown to minimize viscoelastic effects during indentation of polystyrene [28, 29]. Although depth-dependent properties for glassy polymers have been previously reported in the literature [30–34], the methods used here have been shown to provide constant predictions of the material properties in polystyrene throughout the depth, which is observed in our *actual* data set (Fig. 6). Indents on the

aluminum alloy and fused silica used a typical trapezoid loading function, with 5-second loading, holding, and unloading times.

The second set of indents to determine the *actual* values were made on all of the specimens using a technique that allowed for post-processing to correct the surface detection error. In this set, the same preload was used, but after the surface detection, settling time, and thermal drift measurement occurred (i.e. immediately before executing the load function), the indenter was programmed to withdraw from the specimen by 150 nm, move laterally by 1 μm , and reapproach the material and immediately indent in a new location. The offset distance was chosen to ensure that the indents are not affected by any plastic deformation that may have occurred during surface detection. Details of how the indenter was programmed are provided in this document's Supplemental Information document. As the indenter lifts off and performs its second approach, it collects force and displacement data while out of contact with the surface. Once it touches the surface, an increase in load is apparent; however, unlike during surface detection, which uses the criterion of a preload, the data can be post-processed to identify the true location of the surface. Again, 40 indents were performed at each prescribed load with the same loading function on the aluminum alloy and polystyrene, and 20 indents were performed on fused silica.

Since the effect of surface detection error varies with measured contact depth, both sets of indents were performed on the polystyrene for peak loads of 25, 50, 75, 100, 250, 500, 750, 1,000, 2,500, 5,000, 7,500, 10,000 μN , resulting in indents at a range of contact depths, as shown in Fig. 6. Since surface detection error also varies with the applied preload, the experiment was repeated on polystyrene for three preloads: 1, 2, and 5 μN .

The model was also validated on the aluminum alloy with a 2 μN preload over the same set of peak loads, beginning with 100 μN , as lower loads resulted in too small of an indent. On fused silica, a 2 μN preload was used, with indents performed to peak loads of 500, 1000, and 5000 μN .

Post-processing of load-displacement curves to correct for surface detection error

To identify the surface position in the second set of indents, a custom post-processing program was written in *Mathematica* 12. The program identifies the general location of the surface in the data and then uses a combination of nonlinear least-squares fits to the load-displacement curve to automatically identify the surface location. Additional details are provided in the Supplemental Information document.

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Data availability

All data are available in the text. For additional queries, please contact the corresponding author.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

Supplementary Information

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Supplemental Information For
Effect of surface detection error due to elastic-plastic deformation on
nanoindentation measurements of elastic modulus and hardness

Journal of Materials Research

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Supplemental Modeling Results

Blunted Berkovich Indenter

The amount of surface detection error for a blunted Berkovich tip depends on the radius of the spherical portion of the indenter, with a larger radius resulting in smaller errors. The ratio of the *actual* to the *apparent* modulus and hardness values for four material property combinations are given in Fig. S1 as a function of the radius of the blunted end of the indenter. Due to the limited range of radii for Berkovich indenters, typically varying from ~50 nm to a few hundred nanometers, the variance in the amount of error due to the degree of tip blunting is small, relative to other effects such as contact depth. The results in Fig. S1 are plotted for radii of 50 – 250 nm, except in the case of an example material with an *apparent* modulus of $E_r' = 0.1$ GPa, which is given for 100 – 250 nm, to limit the results given here to errors induced by plastic deformation.

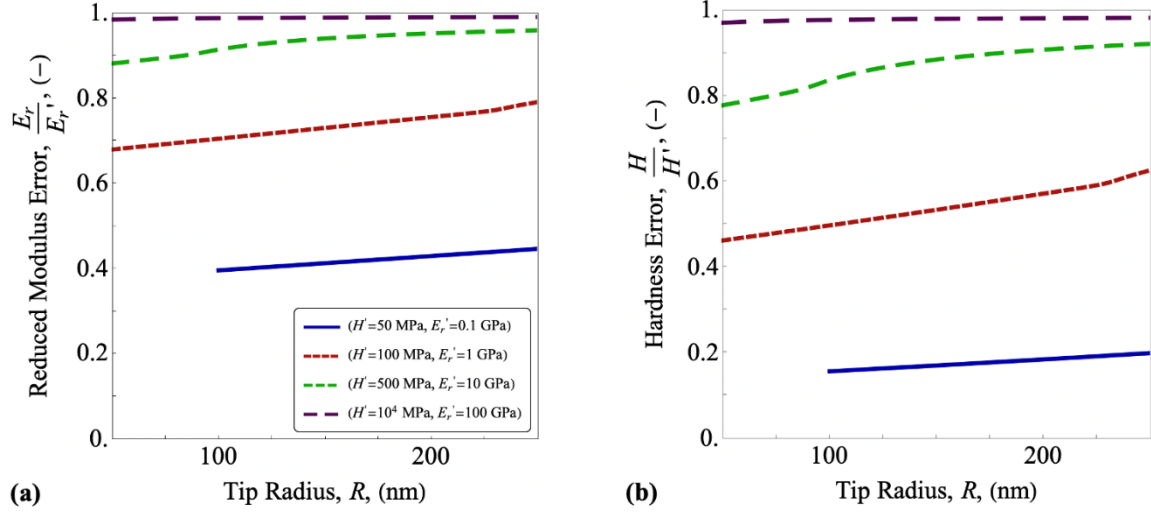


Figure S1: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of the radius of the blunted end of a Berkovich tip is given for four example combinations of *apparent* reduced modulus and hardness, for a preload of 96, nm. For reference, the results for an ideal Berkovich tip for each combination of properties shown here are: respectively. The hardness results are

Spherical Indenter

The amount of error in measuring modulus and hardness with a spherical indenter depends on the *apparent* contact depth and the applied preload. Fig. S2 shows the dependence of the error on the *apparent* contact depth for an ideal spherical indenter with $R = 1 \mu\text{m}$ and $P_0 = 2 \mu\text{N}$. At smaller *apparent* contact depths, the relative size of the surface detection error to the contact depth becomes larger, resulting in significant errors in reduced modulus and hardness calculations.

Similar to the results for the Berkovich tip (Fig. 3), the errors for the spherical tip can be reduced by decreasing the applied preload as shown in Fig. S3. Unlike with the ideal Berkovich model, for a $R = 1 \mu\text{m}$ spherical indenter, it is easier to decrease the preload sufficiently to make the errors resulting from surface detection negligibly small for some material property combinations.

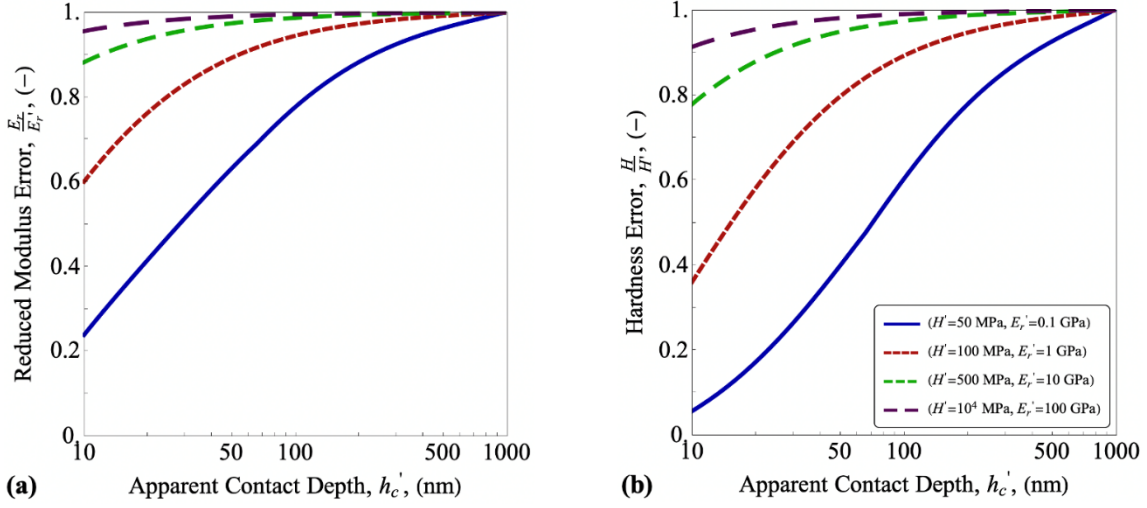


Figure S2: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of the *apparent* contact depth is given for four example combinations of *apparent* reduced modulus and hardness, for an ideal spherical indenter area function of radius $R = 1 \mu\text{m}$ and a preload $P_0 = 2 \mu\text{N}$.

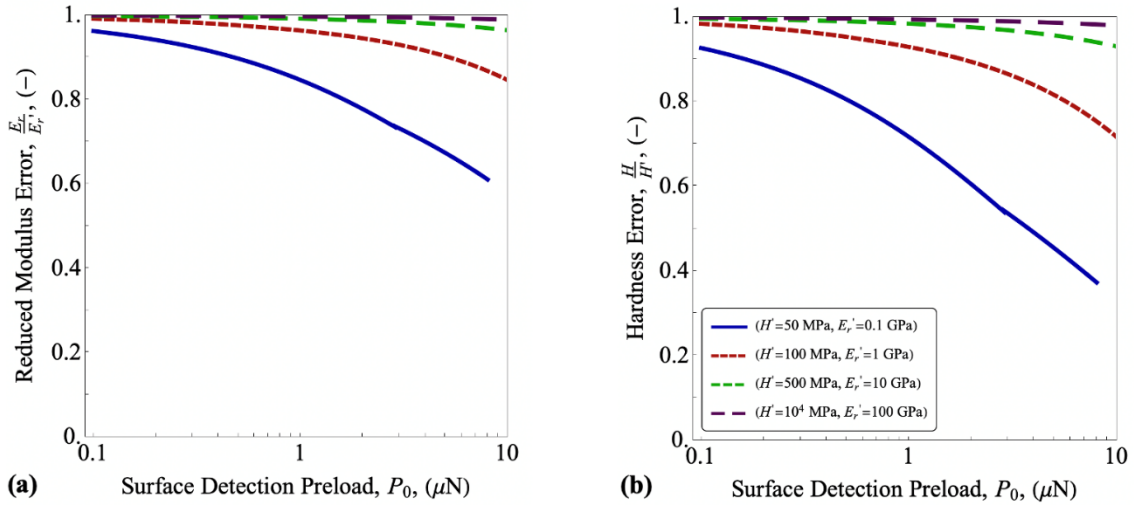


Figure S3: The ratio of the *actual* (a) reduced modulus and (b) hardness to the *apparent* properties as a function of preload, P_0 , is given for four example combinations of *apparent* reduced modulus and hardness for an ideal spherical indenter area function with

Supplemental Information for the Experimental Methodology

Calibrated Area Function

The area function of the Berkovich indenter used in this work was calibrated for depths greater than 20 nm by performing indents on a fused silica specimen with known material properties, over increasing displacements. The results were fit to the following sixth-order polynomial, as described in [2]:

$$A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{\frac{1}{2}} + C_3 h_c^{\frac{1}{4}} + \dots + C_8 h_c^{\frac{1}{128}} \quad (1)$$

where C_i are fitting constants. The area function was calibrated on fused silica between 20 nm and 200 nm, with the value of C_0 fixed at 24.5, which made the area function act as an ideal Berkovich tip for indents deeper than 200 nm. For indents below 20 nm, the area function was determined by fitting a Hertz contact model to elastic indentation load-displacement curves, from which the tip was found to have an approximate radius of 200 nm. A single area function was established for all contact depths by fitting the same sixth order polynomial to the area function calibrated on fused silica for depths greater than 20 nm, and the spherical model for shallow depths. Fig. S4 shows the area function for the indenter used in this work. This area function was used in all calculations in the experimental results in this work.

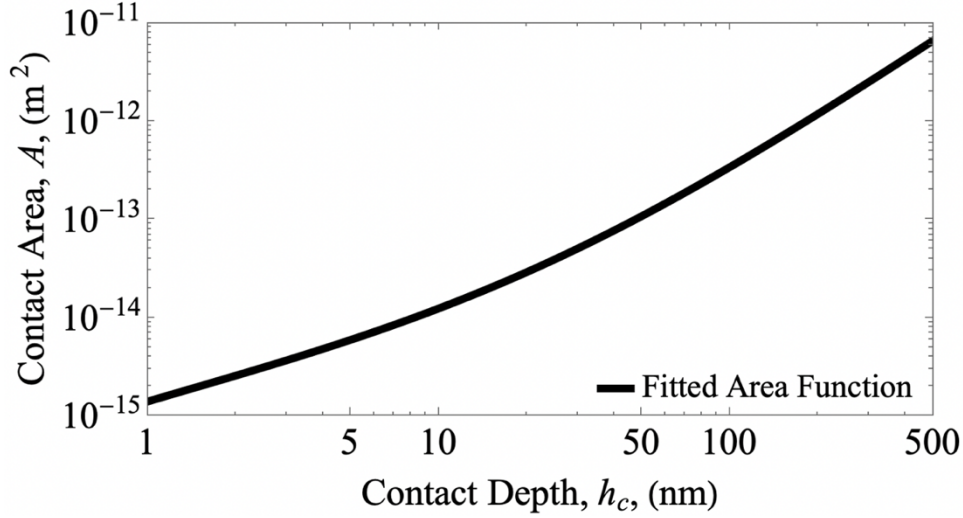


Figure S4: The area function of the indenter used in the experiments.

Contact Imaging of Indents

By fixing the value of $C_0 = 24.5$, the calibrated area function behaves essentially as an ideal Berkovich area function at depths >200 nm. This allowed us to use the same area function for all of our indents, even those beyond the maximum calibration depth. However, to ensure the tip was not contaminated with debris, contact imaging of the deeper indents was performed with the indenter. The deepest indents occurred on polystyrene, with a representative image shown in Fig. S5. It is clear from the topography image (Fig. S5(a)) and the gradient image (Fig. S5(b)) that the tip is pyramidal at the deeper indents. Additionally, the profile in Fig. S5(c) indicates some pileup around the edges of the indent on polystyrene. While pileup does affect the measurements of hardness and elastic modulus in nanoindentation, these effects only become significant when the pileup is sufficiently large relative to the depth of the indent. As noted by *Pharr* [10], another way to estimate the effect of pileup is via the pileup factor h_f/h_m , where h_m is the maximum displacement, and h_f is the final displacement after complete unloading. Clearly, this ratio is bounded between 0 (for a purely elastic indent) and 1 (for a rigid-plastic indent). If this ratio is less

than 0.7, pileup is almost never observed, and values near unity are typically needed for pileup to become significant [10]. Throughout this work, the pileup factor for polystyrene was always near 0.7, suggesting the effect of pileup is small.

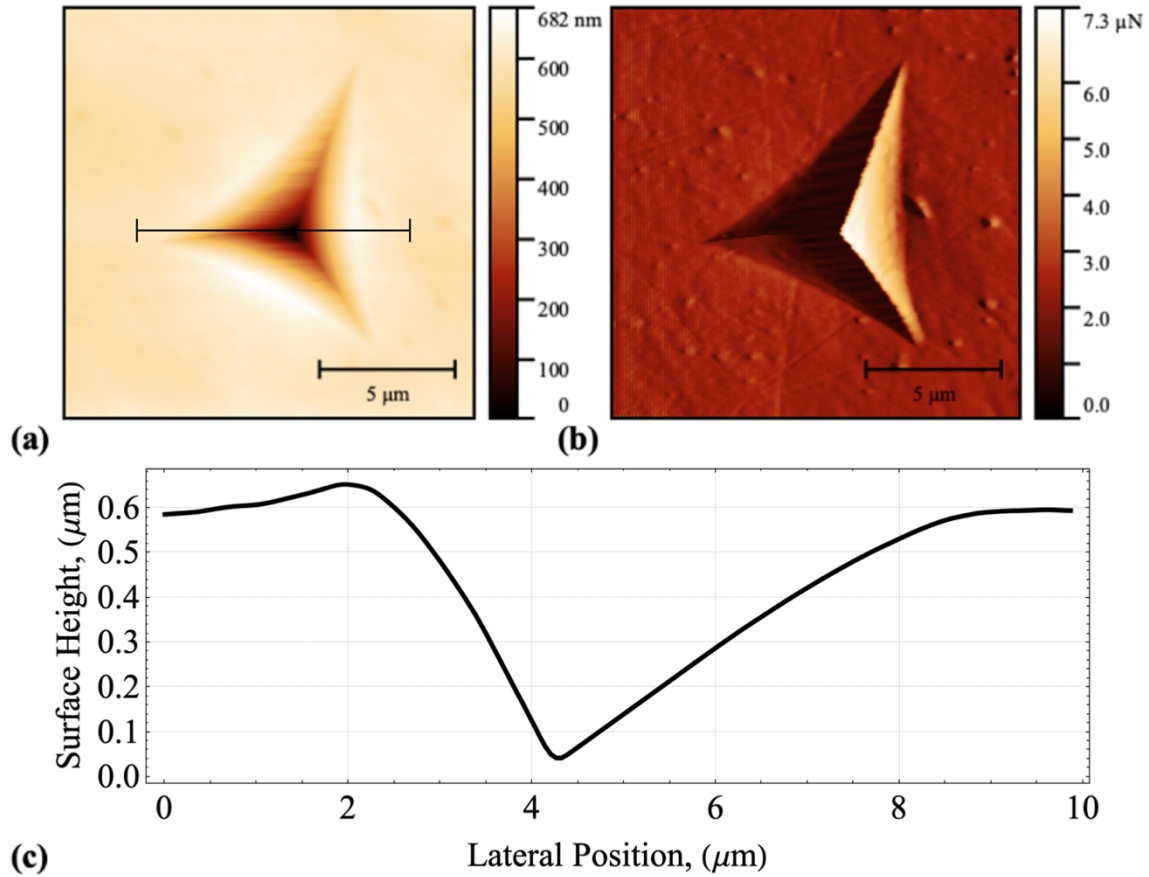


Figure S5: Contact imaging of 5mN peak load residual indent on polystyrene at a setpoint of 2 μN . (a) The forward topography scan with the indenter tip, with a selected contour indicated (b) the forward gradient, and (c) the extracted contour from the topography scan

Representative Loading-Unloading Curves

For indentation on polystyrene, viscoelastic effects can lead to a “nose” on the unloading curve [27]. Representative curves are given in Figure S6(a) for all peak loads, and it is clear that the prescribed loading history (see Methodology) used has minimized this effect.

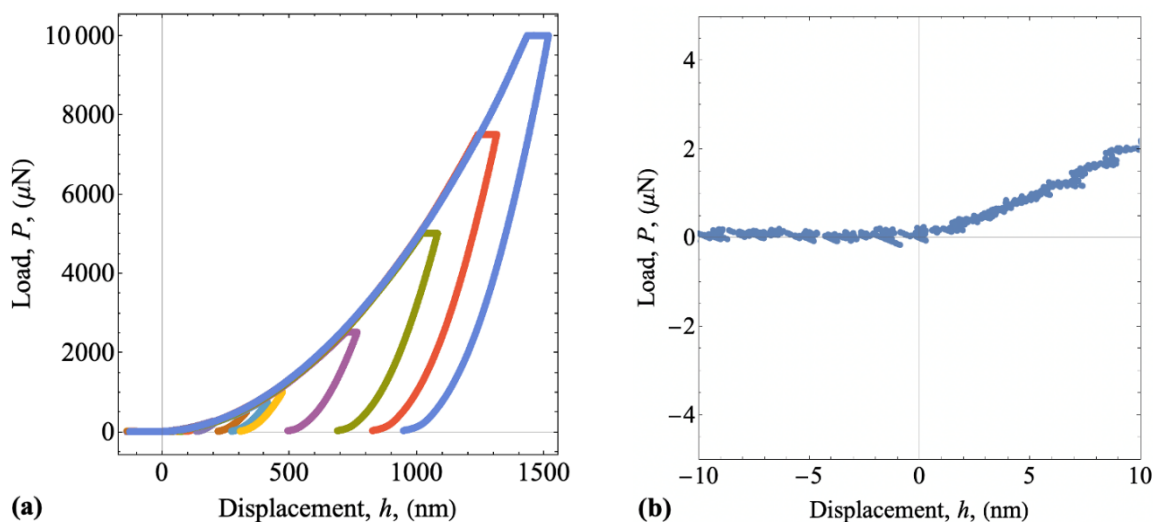


Figure S6: (a) Representative load-displacement curves for each prescribed maximum load from the *actual* dataset on polystyrene are shown. None of the curves demonstrate a rounded “nose” on the unloading curve, which would imply viscoelastic effects. (b) The region of a representative curve from the *actual* dataset showing the surface contact on polystyrene, which shows no significant adhesive effects.

We also verified that there was not significant adhesion in these tests, which can be observed as “snap-in” or “snap-out” in the load displacement curves. The region of a representative loading curve near the surface is shown in Fig. S6(b) to check for snap-in. Clearly, no significant adhesive forces are present. Additionally, no significant snap-out is observed at the ends of the unloading curves (Fig. S6(a)).

Additional representative curves from the aluminum alloy indents are included in Figure S7, which likewise show no extraneous effects that might affect the analysis.

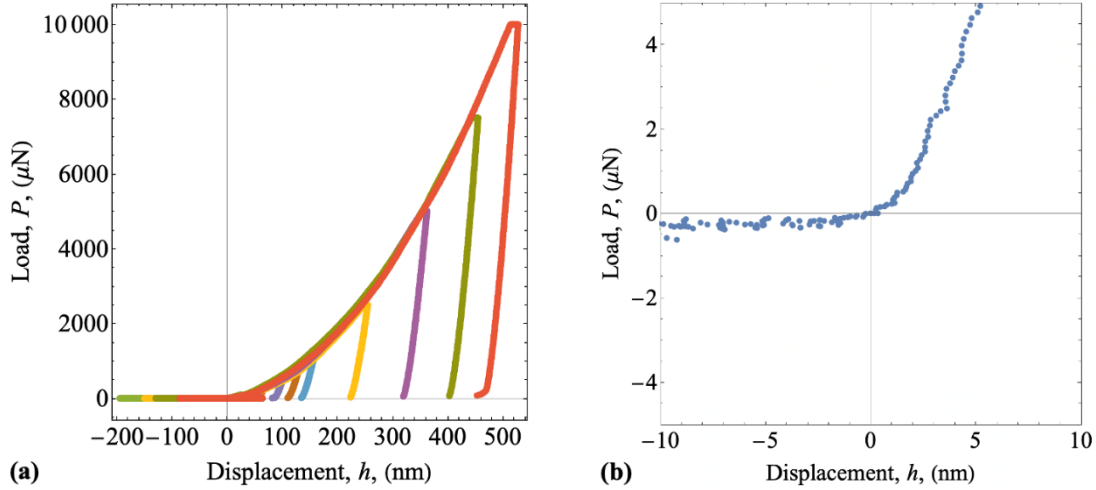


Figure S7: (a) Representative load-displacement curves for each prescribed maximum load from the *actual* dataset on the aluminum alloy. (b) The region of a representative curve from the *actual* dataset showing the surface contact on the aluminum alloy

Performing Liftoff/Offset Indents and Post Processing

For all tests in this work, after surface detection, the tip was held in contact with the specimen for between 125-145 seconds for the motor to settle and for a drift correction measurement to be made. For the *apparent* datasets, the indent then occurred immediately after this pause; however, for the *actual* datasets, after this hold period, we programmed our nanoindenter to retract the tip 150 nm over 10 seconds, move laterally by 1 μm , then reapproach over 10 seconds. For our system, which uses the Hysitron *Triboscan* software, these functions are directly programmable in the load function by prescribing values for the Lift Height, Offset Distance, Lift Time, and Re-seek Time; however, these functions may not be available in other indentation setups, and a different approach would be required.

After the *actual* data was collected, it needed to be post-processed to correctly identify the surface and remove the surface detection error. This could be performed by varying methods, but for this work we developed a program in *Mathematica 12* to automatically perform the process, as illustrated in Fig. S8. The program first identifies the general location of the surface at the end of

the in-air data and beginning of the loading curve and only considers the data in that region (Fig. S8(b)). We note here that in the lift-off and offset loading curves the loads may be lesser or greater than the preload at zero displacement. This is a result of surface roughness/non-flatness and/or specimen tilt, such that the offset indent location has a different surface height than the location where the surface detection occurred. For example, if the surface in the offset location is higher than in the surface detection region, the tip encounters the surface earlier as it reapproaches the specimen and thus registers a greater load than the preload when it returns to the zero displacement location. An example of this effect is evident in Fig. S8(b). This loading curve generally has the form of a power-law that can be fit to identify the surface location, but we found determining the surface based on this approach to be prone to errors and used the alternate method described below.

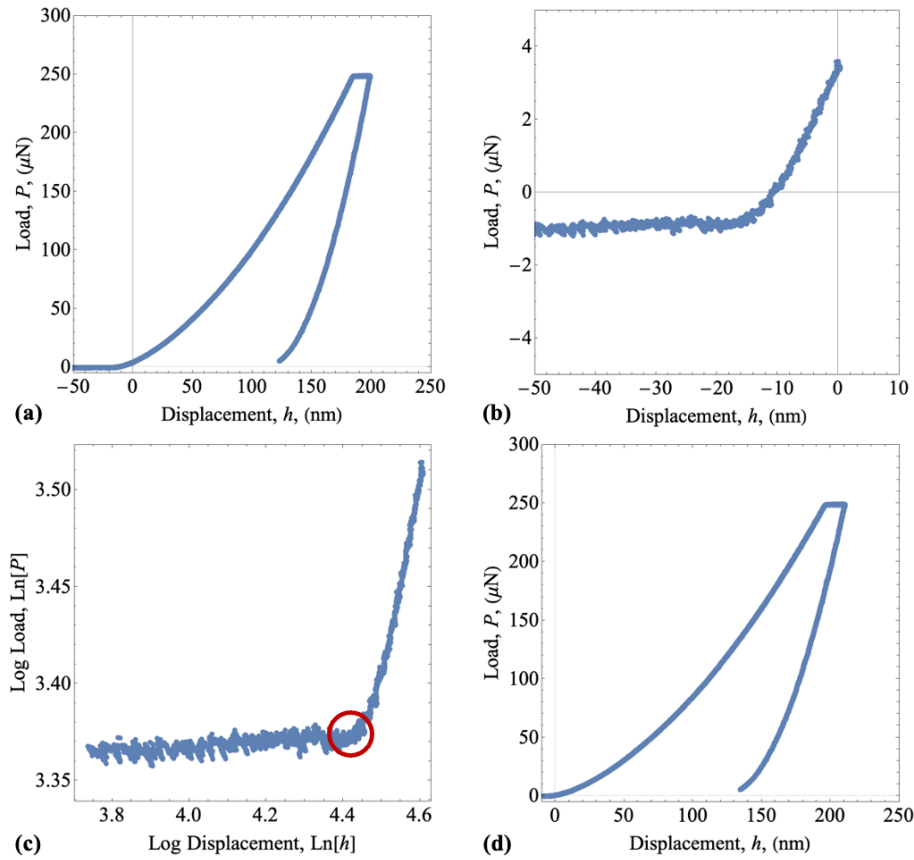


Figure S8: (a) Representative *actual* load-displacement curve before post processing. (b) Isolating the region of the surface in the in-air and loading curve. (c) Shifting the data positive and taking the logarithm before fitting two lines to the resulting curve to identify the surface location (d) Resulting curve with the surface location shifted to the origin.

Instead, the program employs an algorithm that creates a new shifted dataset, such that all values are positive, and then takes the natural log of both load and displacement. This results in the data near the contact point resembling an intersection between two straight lines, a flat line representing the in-air data, and a sloped line representing the loading curve (Fig. S8(c)). A non-linear least squares fit is used to fit a piecewise function of two lines to this curve, with the intersection being the surface location. This point is then extrapolated back to the original data set and this point is shifted to the origin.

A non-obvious detail of the nanoindenter used in this work is that the first data point after surface detection is taken to be both zero displacement (which results in surface detection error) and zero load, despite the fact that the force at the first data point is actually at the preload value. This results in a vertical shift in the data so that the in-air data registers as negative load (see Fig. S8(b)) and the peak load applied during indentation is less than that specified in the loading sequence, with the difference being equal to the preload value. This error in load is automatically corrected for in the post-processing of the *actual* data, as shown in Fig. S8, such that the in-air data is measured as zero load, and the peak load is identical to what was programmed. We also correct the *apparent* data for this error in load, such that the peak load matches what was programmed and achieved.