3. Surface Mechanical Probing of Materials

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Surface mechanical properties are of immense importance for many technological applications. In particular, material property measurements at the submicron, such as the surface modulus, the hardness, the scratch resistance, the fracture toughness and fatigue properties are essential for the characterization of miniaturized electronic, optical, mechanical, and biomedical devices. In this chapter we will address two aspects of surface mechanical probing of materials: hardness measurements and elastic modulus determination.

3.1 Hardness determination

Hardness implies the resistance to local deformation. Depending on the material hardness implies different things: For instance, hardness is a measure of the indentation depth for a plastic material (i.e., a solid material that is indented beyond its yield point). For a brittle material, hardness is defined in the course of scratch experiment. Scratch tests reveal the oldest form of hardness measurements, typically referred to as Mohs' Hardness. More familiar we are today with the quasi-static indentation hardness method by Tabor. A third method is the rebound or dynamic hardness method. It involves a diamond-tipped hammer that is dropped from a fixed height onto the test surface. The hardness is expressed in terms of the energy of the impact and the size of the remaining indentation.

3.1.1 Quasi-static indentation hardness method

The indentation hardness is essentially a measure of the sample's plastic deformation properties. The method depends on the time of loading, the temperature and other operating conditions (e.g., humidity). Typically a spherical, conical or pyramidal indenter is forced into the surface. The ratio between a corresponding load, $L$, and a characteristic indentation area ($residual area A_r$) defines the hardness number in GPa (or kg/mm$^2$); i.e.,

$$H_r = \frac{L}{A_r}.$$  \hspace{1cm} (1)

The hardness number is named based on indenter shape. If $A_r$ is a curved surface then one used either a Brinell, Rockwell or a Vickers (4 sided pyramid) hardness tester. $A_r$ is taken as the projected area for a Knoop (4 sided pyramid) or Berkovich (3 sided pyramid) hardness tester.

It is generally accepted that the depth of indentation should never exceed 30% of the film thickness of a coating because the hardness number is affected by the substrate. This rule of thumb is however strongly dependent on the tip geometry. For thin films a sharp tip is desirable. Highly pointed tips are found in Berkovich and Vickers hardness testers.
A indenter consists of a driver, a force sensor, and a sharp tip with well defined geometry. The drivers and force sensor have been improved significantly over the last 20 years. As drivers serve

- magnetic moving coil systems (see Fig. 1), and
- piezo electric devices.

![Fig. 1 Schematic of a Nanoindenter with magnetic moving coil system (Source: Micro/Nanotribology, ed. B. Bhushan, CRC)](image1)

![Fig. 2 Schematic of a Nanoindenter with double leaf spring strain gauge (Source: Micro/Nanotribology, ed. B. Bhushan, CRC)](image2)

As force sensors serve

- strain gauges with double leaf spring (Fig. 2)
- capacitance sensors

The requirements for indenters are

- high elastic modulus (i.e., not very compliant)
- no plastic deformation of the tip
- low friction (accomplished by the tip geometry)
- well defined geometry with smooth surfaces

In order to obtain accurate hardness values the examined surface should exhibit also low roughness. Diamond as tip material provides high elastic modulus, no plastic deformation, low friction and smooth crystal surfaces.
3.1.2 Berkovich Indenter

The tip is a three-sided pyramid and provides a sharply pointed tip (mathematically intercept at a single point). A sharply pointed tip ensures localized indentation. The wide opening angle (blunt tip) is desirable to avoid cutting.

![Ideal Berkovich tip (schematic)](Fig. 3(a): Ideal Berkovich tip (schematic). The Berkovich tip is a blunt tip with an opening angle of 76.9 and 65.3 degrees, but with a sharp contact. In contrast to it is the AFM tip. The SFM tip has an acute opening angle but often exhibits a dull contact. (b) SEM image of an actual tip. (Source: Micro/Nanotribology, ed. B. Bhushan, CRC)

Nanoindentations are difficult to calibrate as the tip shape of the Berkovich tips is not well defined (see Fig. 3b). Typically the radius of the tip is < 0.1 µm, and indentation are performed of 10 nm to 20 µm depth. The projected contact area can be calculated, based on the tip only, as

\[ A = 0.433a^2 = 23.76h^2 \]  

where \( a \) is the tip triangular base side, and \( h \) is the tip length. This contact area is only related to the actual indentation if the material is fully plastic. The deformation pattern of an elastic-plastic sample indentation exhibits the following parameters (see also Fig. 4):

- \( h_f \): residual harness impression (after elastic recovery)
- \( h_c \): contact depth (i.e., depth the indenter is actually in contact with material)
- \( h_{\text{max}} \): calculated (projected) indentation at the peak of the load
- \( h_s \): depression of the sample around the indentation; i.e., \( h_s = h - h_c \)
- \( h_p \): slope extrapolated indentation depth reminder; i.e.,

\[ h_p = h_{\text{max}} - \frac{L_{\text{max}}}{S_{\text{max}}} \]

\( S_{\text{max}} \) is the stiffness (inverse compliance) of the sample only, which corresponds to the slope of the unloading curve at the maximum load. For a Berkovich indenter \( h_c > h_p \) (\( h_c = h_p \) only for a flat punch geometry). The contact area for Berkovich tips is approximated as
which replaces equation (2) for elastic-plastic indentation. Equation (3) has been derived for a Vickers hardness tester. Finally the Berkovich hardness is defined as

\[ H_B = \frac{L_{\text{max}}}{A} \]  

Based on the degree of indentation one can classify indentations into elastic, fully plastic and elastic-plastic (Fig. 5).
3.1.3 Area Function

The relationship between the area and the system compliance is called the area function. The total compliance (compliance = deformation per unit force) during an indentation experiment for two spring system in series is

\[ C = C_i + C_s \]  

(5)

where \( C_i \) and \( C_s \) are the compliances of the indenter and the sample, respectively. The compliance for a Vickers, Knoop, and Berkovich indenter is:

\[ C_s = \frac{1}{S_{\text{max}}} \cdot \frac{dh}{dL} \approx \frac{1}{2E_r} \left( \frac{\pi}{A} \right)^{\frac{1}{2}} \]  

(6)

derived by King (1987), where \( S \) is the slope of the unloading curve at maximum load (see above) and the residual elastic modulus, \( E_r \), (assumed to be a constant) is defined as

\[ E_r = \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_i^2}{E_i} \]  

(7)

with the elastic moduli and Poisson ratios \( E_s, E_i, \nu_s \) and \( \nu_i \) of the sample and the indenter (diamond: 1140 GPa and 0.07). Hence, equations (5) and (6) yield a linear relationship of the compliance with \( A^{-1/2} \), i.e.,

\[ C = C_i + \frac{1}{2E_r} \left( \frac{\pi}{A} \right)^{\frac{1}{2}} . \]  

(8)

3.1.4 Indentation Procedure

Classical Procedure: Make a series of indentations at various depths (predominantly plastic). Measure the size of the indentation by direct imaging (TEM carbon replicas, or even better by SFM). It is observed that the ideal geometry underestimates the contact area, which leads to overestimation of the hardness and elastic modulus, especially at small indentation depths.

Procedure for Nanoindentation: Oliver and Pharr (1992) proposed an easier method for determining the area function that requires no imaging. Their procedure is based on the assumption that the Young's modulus is independent of the indentation depth.

Procedure:
- Calibrate the indenter with two standard materials that are elastically isotropic, their moduli well known, and their moduli depth independent:
  - aluminum (soft material, provides deep indentations), \( E_i = 70.4 \text{ GPa}, \nu_i = 0.347 \);
  - fused quartz (hard material, provides shallow indentations) \( E_i = 102 \text{ GPa}, \nu_i = 0.17 \).
(a) **Determine $C_i$ with aluminum** by using equation (8) and setting $A = 24.5h_c^2$. This area function assumes a perfectly sharp indenter (ideal Berkovitch). Use typical high indentation load series between (700-4000 nm indentation depth).

(b) **Determine the area function of the realistic Berkovich tip with fused quartz** with minimum contact depths (15-700 nm) by rewriting equation (8) as

$$A = \frac{\pi}{4} \frac{1}{E_c^2} \frac{1}{(C - C_i)} \approx 24.5(h_c)^2 + C_1h_c + C_2(h_c)^{1/2} + C_3(h_c)^{1/4} + \ldots + C_8(h_c)^{1/128}$$

and fit to an eighth order polynomial.

Nanoindentation measurements are provided in Figure 7(a+b) for thin films. It was found that adhesive forces and surface roughness decrease the elastic modulus with increased contact area.

![Figure 6a: Single crystal aluminum, soft, not much elastic recovery](image)

![Figure 6b: Single crystal sapphire, hard, significant elastic recovery.](image)

![Figure 7a: Load dependence of hardness](image)

![Figure 7b: Load dependence of elastic moduli](image)

*Fig. 6a: Single crystal aluminum, soft, not much elastic recovery*

*Fig. 6b: Single crystal sapphire, hard, significant elastic recovery.*

*Fig. 7a: Load dependence of hardness Source: *Micro/Nanotribology*, ed. B. Bhushan, CRC*

*Fig. 7b: Load dependence of elastic moduli Source: *Micro/Nanotribology*, ed. B. Bhushan, CRC*
3.2 Indentation Mechanics (by Scott Sills)

The following sub-sections will focus on characterizing the phenomena portrayed during indentations in materials which exhibit different stress-strain and strain rate relationships.

3.2.1 Elastic-Perfectly Plastic

During normal indentation of an elastic-plastic material, when the yield point of the more ductile material is first exceeded, the onset of plastic deformation commences. Initially, the plastic region is small and completely contained by the surrounding elastic material. Hence, the plastic strains are of the same order of magnitude as the surrounding elastic strains. The plastically displaced material is fully accommodated by elastic expansion of the surrounding solid. This is referred to as the “constrained deformation” regime because the flowing or plastically deforming volume is fully constrained by the surrounding elastic medium. As the applied strain is increased, i.e. increased load or sharper indenter, a greater pressure beneath the tip is required to produce the necessary expansion. Eventually, sufficient pressure is achieved where the plastic region reaches the free surface, allowing the displaced material to escape via “uncontained” plastic flow along the sides of the indenter.

The onset of plastic yield, or constrained deformation, is assessed by applying an appropriate yield criterion. The two most commonly applied criteria are the Tresca’s maximum shear stress criterion, where yielding occurs when the maximum shear stress, or half the difference between the maximum and minimum principle stresses, reaches the yield stress in pure shear or half the yield stress in simple compression (or tension):\(^1\)

\[
\max \left[ \frac{1}{2} |\sigma_1 - \sigma_2|, \frac{1}{2} |\sigma_2 - \sigma_3|, \frac{1}{2} |\sigma_3 - \sigma_1| \right] = k = \frac{Y}{2},
\]

(4.3.1-1)

and the von Mises’ shear strain-energy criterion, where yielding occurs once the deformation energy equals the deformation energy at yield in simple compression or pure shear.\(^2\) Therefore, by the von Mises criterion, yielding occurs when the square root of the second invariant, \(J_2\), of the stress deviator tensor, \(S_{ij}\), reaches the yield stress in simple shear or \(1/\sqrt{3}\) of the yield stress in simple compression:

\[
\sqrt{J_2} = \left[ \frac{1}{2} S_{ij} \right]^{1/2} = \left\{ \frac{1}{6} \left[ (\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 \right] \right\}^{1/2} = k = \frac{Y}{\sqrt{3}}
\]

(4.3.1-2)
where $\sigma_1$, $\sigma_2$, and $\sigma_3$ are the principle stresses in the state of complex stress, and $k$ and $Y$ represent the yield stresses in pure shear and simple compression (or tension), respectively. For detailed analysis of the state of complex stress and formulation of the yield criteria, the reader is referred to $^3$.

Experiments on isotropic metals support the von Mises criterion over Tresca’s; however, the discrepancy between the two is relatively small considering the variability in $k$ or $Y$ and the inherent anisotropies in most materials. Therefore, it is generally acceptable to apply Tresca’s criterion for its mathematical simplicity.

Relating the stresses from the yield criteria to the mean contact pressure under the indenter,

$$p_m = cY$$

(4.3.1-3)

where, for the onset of constrained plastic yield, $c$ has a value of about 0.5 for conical indenters, and may vary depending on the indenter geometry and the friction at the interface.$^1$

The onset of unconstrained plastic flow, i.e. the point when the plastic yield zone reaches the free surface, is expected to occur when the contact pressure reaches the yield stress given by rigid-plastic theory. Based on a number of numerical analyses and indentation measurements with rigid spheres and cones in elastic-plastic half-spaces, Johnson has determined a value of $c \approx 2.8$, in equation 3.

In the transitional regime, when the contact pressure lies between $0.5 \sim 3Y$, plastic flow is contained by the surrounding elastic material. The resulting deformation is generally in the form of radial expansion with roughly hemispherical contours of equal strain.$^1$ Based on these observations, Johnson (1970)$^4$ has developed a simple cavity model of elastic-plastic indentation, Figure 4.3.1-1. The cavity model assumes that directly beneath the indenter contact surface, a hemispherical core with a radius equal to that of the projected contact area, $a$, has a hydrostatic stress component equal to the mean contact pressure, $p_m$. Immediately beyond the core lies the plastic zone, and at the core-plastic boundary, the radial stress component in the plastic zone equals the hydrostatic stress of the core.

![Figure 4.3.1-1: Cavity model elastic-perfectly plastic indentation by a cone.](image)
Within the plastic zone, stresses and displacements have radial symmetry, and the plastic strains gradually diminish with increasing radial distance, until they match the elastic strains at the elastic-plastic boundary, $c (c>a)$.

Based on Hill, the stresses in the plastic zone, $a \leq r \leq c$, are characterized by:

$$\frac{\sigma_r}{Y} = -2\ln\left(\frac{c}{r}\right) - \frac{2}{3}, \quad \frac{\sigma_\theta}{Y} = -2\ln\left(\frac{c}{r}\right) + \frac{1}{3}$$  \hspace{1cm} (4.3.1-4a,b)

and in the elastic zone, where $r \geq c$

$$\frac{\sigma_r}{Y} = -\frac{2}{3}\left(\frac{c}{r}\right)^3, \quad \frac{\sigma_\theta}{Y} = \frac{1}{3}\left(\frac{c}{r}\right)^3$$  \hspace{1cm} (4.3.1-5a,b)

At the core-plastic boundary, the core pressure is given by:

$$\frac{p_m}{Y} = 2\ln\left(\frac{c}{a}\right) + \frac{2}{3}$$  \hspace{1cm} (4.3.1-6)

Equation 6 implies that the elastic-plastic boundary coincides with the core-plastic boundary, $c=a$, at $p_m=\gamma Y$, and at reduced pressures, no plastic flow occurs. Therefore, the cavity model predicts that the onset of plastic yield occurs at $p_m=\gamma Y$ which is close to the value of $c \approx 0.5$ reported by Johnson (1970). The difference is attributed to $\beta$, and friction at the interface.

Radial displacement of matter at the core-plastic boundary, $r=a$, during an increment of penetration, $dh$, must accommodate the volume of material displaced by the indenter. Neglecting core compressibility, conservation of core volume requires:

$$2\pi a^2 du(a) = \pi a^2 dh = \pi a^2 \tan(\beta) da$$  \hspace{1cm} (4.3.1-7)

The radial displacements within the plastic zone are given by (Hill 1950):

$$\frac{du(r)}{dc} = \frac{Y}{E} \left[ 3(1-\nu)\left(\frac{c}{r}\right)^2 - 2(1-2\nu)\left(\frac{r}{c}\right) \right]$$  \hspace{1cm} (4.3.1-8)
Equations 7 and 8 are used to locate the elastic-plastic boundary, \( c \), recognizing that for a conical indenter, geometrical similarity of the strain field with continued penetration requires \( \frac{dc}{da} = \frac{c}{a} = \text{constant} \):

\[
\left( \frac{c}{a} \right)^3 = \frac{1}{6(1-\nu)} \left[ \frac{E \tan \beta}{Y} + 4(1-2\nu) \right] 
\]

(4.3.1-9)

The core pressure is determined via substituting \( \frac{c}{a} \) into equation 6, and for an incompressible material, i.e. \( \nu = 0.5 \):

\[
\frac{p_m}{Y} = \frac{2}{3} \left[ 1 + \ln \left( \frac{1}{3} \frac{E \tan \beta}{Y} \right) \right] 
\]

(4.3.1-10)

The hydrostatic core pressure appears solely dependent on the parameter \( \frac{(E/Y)\tan \beta}{Y} \), which represents the ratio of the strain imposed by the indenter (\( \tan \beta \)) to the strain capacity of the indented material \( \frac{Y}{E} \). The indentation pressure under elastic, elastic-plastic, and fully plastic conditions is generally correlated as dimensionless contact pressure, \( P_m/Y \), versus dimensionless strain, \( \frac{(E/Y)\tan \beta}{Y} \).

### 3.2.2 Elastic-Real Plastic

The above analysis was limited to elastic-perfectly plastic materials with a constant yield stress; however, Tabor (1951) has shown that the analysis for a perfectly plastic solid may be applied, with good approximation, to work (or strain) hardening materials if \( Y \) is replaced by a “representative” flow stress \( Y_R \), measured in simple compression at a representative strain \( \varepsilon_R \), where for a material whose strain hardening is described by a power law relation:

\[
Y_R = \sigma_o \left( \frac{\varepsilon_R}{\sigma_o} \right)^n 
\]

(4.3.2-1)

where \( n \) is the reciprocal of the work hardening index, and \( \sigma_o \) is the work hardening coefficient. Substituting into equation 3 and using a value of \( c \) corresponding to perfect plastic yielding, i.e. 3:

\[
\varepsilon_R = \varepsilon_o \left( \frac{p_m}{3\sigma_o} \right)^n 
\]

(4.3.2-2)

Equation 12 enables \( \varepsilon_R \) to be determined given the appropriate expression for \( p_m \), and for a conical indenter, is approximated by:

\[
\varepsilon_R = 0.2 \tan \beta 
\]

(4.3.2-3)
3.2.3 Thermoelastic

The analysis of this thermoelastic contact problem consists of three parts: (i) heat transfer analysis to determine the temperature distribution within the two contacting bodies; (ii) analysis of the thermal expansion of the bodies to determine the thermal distortion of the contact area; and (iii) analysis of the isothermal contact problem to assess the contact stresses resulting from the deformed profile. With a conical indenter, a symmetrical body of revolution, we will only be concerned with the two dimensional case ($z$ and $r$ in cylindrical coordinates). Assuming that the indenter reaches its final hot temperature before the polymer reaches $T_g$ (when significant penetration begins), the problem may be formulated as follows: a hot body at an initial uniform temperature $T_H$ is pressed into contact with a colder body at initial uniform temperature $T_C$, and the interfacial temperature, $T_i$, is uniform throughout the contact region. The contact area is circular with radius $a$. Initially, we will assume a frictionless interface. The distribution of heat flux, $\dot{q}$, across the interface is given by:

$$\dot{q}(r) = \frac{2k_H (T_H - T_i)}{\pi(a^2 - r^2)^{1/2}} = \frac{2k_C (T_i - T_C)}{\pi(a^2 - r^2)^{1/2}} \quad (4.3.4-1)$$

where $k_i$ is the thermal conductivity of the respective body. Integrating over the contact area gives the total heat flux, $\dot{Q}$:

$$\dot{Q} = 4k_H a(T_H - T_i) = 4k_C a(T_i - T_C) = 4\bar{k} a(T_H - T_C) \quad (4.3.4-2)$$

where $\bar{k} = k_H k_C / (k_H + k_C)$. The surface profile resulting from the thermoelastic distortion is then determined by:

$$\bar{u}_z(r) = \frac{2}{\pi} e\bar{k} a \left( T_H - T_C \right) \left[ \ln \left( \frac{r_o}{a} \right) - \ln \left( 1 + \left( 1 - \frac{r^2}{a^2} \right)^{1/2} \right) \right] + \left( 1 - \frac{r^2}{a^2} \right)^{1/2} \quad r \leq a \quad (4.3.4-3a)$$

$$\bar{u}_z(r) = \frac{2}{\pi} e\bar{k} a \left( T_C - T_o \right) \ln \left( \frac{r_o}{r} \right) \quad r > a \quad (4.3.4-3b)$$

where $c = (1+\nu)\alpha/k$ is referred to as the distortivity of the material, $\nu$ is the Poisson ratio, $\alpha$ is the coefficient of thermal expansion, and $r_o$ is the position on the surface where $\bar{u}_z = 0$. The distorted surface profile affects the contact area and thus the contact pressure. The thermally induced pressure component may be expressed as:
\[ p_t(r) = (c_2 - c_1) \frac{\dot{E}^*}{2 \pi^2 a} \left\{ \frac{\pi^2}{8} - X_2 \left[ \frac{a - \left( a^2 - r^2 \right)^{1/2}}{a + \left( a^2 - r^2 \right)^{1/2}} \right] \right\} \quad (4.3.4-4) \]

where \( E^* \) is the reduced modulus and \( X_2 \) is Legendre’s Chi function, i.e.

\[ X_2(x) = \frac{1}{2} \int \ln \left( \frac{1 + s}{1 - s} \right) ds = \sum_{m=1}^{\infty} \frac{x^{2m-1}}{(2m-1)^2} \quad (4.3.4-5) \]

The thermal component to the mean contact pressure is given by:

\[ P_t = \frac{1}{2\pi} (c_2 - c_1) \dot{E}^* a = \frac{2}{\pi} k (T_H - T_C) E^* a^2 \quad (4.3.4-6) \]

The net pressure, corresponding to the total load, is found by adding the thermal component to the isothermal contact pressure. For a Hertzian contact pressure it is:

\[ P = P_t + P_{iso} = P_t + \frac{4E^* a^3}{3R} \quad (4.3.4-7) \]

The total load, \( P \), provides the following relationship:

\[ \beta \left( \frac{a}{a_o} \right)^2 + \left( \frac{a}{a_o} \right)^3 = 1 \quad (4.3.4-8a) \]

with

\[ \beta = \frac{3kR}{2a_o} (c_2 - c_1)(T_1 - T_2) \quad (4.3.4-8b) \]

and \( a_o = \left( \frac{3RP}{4E^*} \right)^{1/3} \) assuming an isothermal Hertzian contact. From equation 8a, it may be inferred for \( \beta > 0 \), that an increase in the differential distortivity or temperature difference causes the relative curvature of the two surfaces to increase and the contact area, \( a \), to decrease.$^1$

**Viscoelastic**

Many materials, notably polymers, exhibit viscoelastic behavior, which is characterized by a time and temperature dependent stress-strain relationship. In the context of non-conforming point contact, such as indentations, the issue becomes one of determining the variation with time of the contact area and pressure distribution resulting from a prescribed loading or penetration schedule. In cases where the corresponding solution for a purely elastic material is known, the simplest approach to this problem,
based on Radok (1957), consists of replacing the elastic constant in the elastic solution with the corresponding integral operator from the viscoelastic stress-strain relations, i.e. the creep compliance or relaxation functions.\(^1\)

### 3.3 Rim Formation During Indentation (by Scott Sills)

During indentation of a rigid plastic solid, the displaced material appears in the piled-up rim around the periphery of the indentation site; but with elastic-plastic materials, most, if not all, of the displaced material is accommodated by radial expansion of the elastic surroundings, with an imperceptible change in the surface dimensions of the indented material.

Briscoe et al. have found for indentation and scratch hardnesses studies of PMMA, the measured yield stress is strongly dependent on both the indenter geometry (related to strain) and applied strain rate. During normal indentation with conical indentors of large excluded angles (\(\beta=75^\circ\)), PMMA deforms by extrusion to the free surface with the creation of a piled-up rim. With blunter cones, i.e. lower \(\beta\), deformation occurred elastoplasticly resulting in little or no pile-up.\(^6\) In nano-scratch studies on PMMA, Adams et al. have demonstrated that the height of the pile-up will increase with \(\tan\beta\).\(^7\) In a similar study on polycarbonate, Jardret et al. have observed distinctly different pile-up formations during nano-scratching on samples of identical hardness. The difference in pile-up height was attributed to the strain magnitude, with an increased rim height for larger strains, i.e larger \(\tan\beta\).\(^8\) With a 2D finite element approach, Ramond-Angélélis has modeled piled-up rim formation in viscoelastic-perfectly plastic materials as a function of the rheological factor \(X\), defined as ratio between the strain imposed by the indenter (\(\varepsilon\sim\tan\beta\)) and the strain capacity of the material (\(\varepsilon\sim Y/E\)), i.e. \(X = E/Y \tan\beta\). These results are presented in Figure 4.4-1, and suggest for values of \(X < \sim 10\), the deformation during indentation is mainly elastic with little pile-up, and for \(X > \sim 100\), the deformation is primarily plastic resulting in substantial pile-up.\(^9\)

Pile-up formation in viscoelastic materials is also dependent on the strain rate. At high strain rates, PMMA displays a noticeable strain softening behavior that becomes more pronounced with increasingly higher strain rates and is absent at reduced strain rates (\(<\sim 10^{-5}/s\)).\(^6\) Consequently, the yield stress decreases with increased strain rate, and is accompanied by the appearance of shear bands, which has been attributed to the onset of pile-up.\(^10\), \(^11\) The effect of a high strain rate at large strains, i.e. high \(\tan\beta\), may also induce adiabatic heating within the shear bands, which would promote large inhomogeneous strains, as well as, enhanced strain softening.\(^6\)

Pile-up is also influenced by the strain hardening behavior of the material. A large capacity for strain hardening advances the plastic zone further into the material, thus decreasing pile-up adjacent to the indenter.\(^12\) For an elastic-plastic material which strain hardens according the following power law:

\[
Y_R = \sigma_0 \left( \frac{\varepsilon_R}{\varepsilon_0} \right)^\frac{1}{n}
\]

\[(4.4-1)\]
where \( n \) is the reciprocal of the work hardening index, and \( \sigma_o \) is the work hardening coefficient, Matthews has proposed the indenter penetration depth, \( h \), follows: \(^{12}\)

\[
\frac{\xi}{h} = \frac{1}{2} \left( \frac{2n + 1}{2n} \right)^{2(n-1)} - 1
\]

(4.4-2)

where \( \xi \) represents the vertical dimension with respect to the neutral surface of a piled-up rim or sunken depression at the periphery of the indentation, Figure 4.4-2.

\[ \text{Figure 4.4-1: Cross-sectional view of elasto-plastic indentation under load and after unloading for a rheological factor, } X, \text{ ranging from 1-1000. Results obtained by Ramond-Angélélis with a 2-D finite element model.}^{9} \]

\[ \text{Figure 4.4-2: Peripheral deformation for opposing extremes of stress sensitivity. Sunken depression observed on materials that exhibit strong strain hardening, and piled-up rims are observed on materials that exhibit slight strain hardening.} \]
For a conical indenter:

\[ h + \frac{\xi}{a} \tan \beta \]  \hspace{1cm} (4.4-3)

Combined with equation 4.4-2, the indentation depth becomes:

\[ h = 2 \left( \frac{2n}{2n+1} \right)^{2(n-1)} a \tan \beta \]  \hspace{1cm} (4.4-4)

For \( h/(\tan \beta) > 1 \) (i.e. \( n < 3.8 \)), a sunken depression is formed, and when \( n \) exceeds 3.8, \( h/(\tan \beta) < 1 \) and a piled-up rim is formed.

In viscoelastic materials, such as polymers, strain hardening must also be addressed. The pile-up formed during the nano-scratch studies on PMMA was analyzed by Adams et al. Indentation hardness measurements of the piled-up rim revealed a maximum hardness value at the rim apex, which decayed asymptotically to the hardness of the unperturbed film with increasing distance from the indentation site. Both the rim height and the extent of strain hardening within the pile-up were increased with the applied strain, i.e. \( \tan \beta \). In addition, Adams et al. observed that the hardened pile-up of an existing scratch reduces the depth of a subsequent parallel scratch within the strain hardened area. This is important in the context of thermomechanical data storage where ultra high storage densities are sought by minimizing the pitch of indented data-bits. A tight indentation pitch with overlapping strain hardened zones would likely result in non-uniform indentation depths, sacrificing device reliability.

The above discussion has been limited to bulk materials, and the formation of piled-up rims has been attributed primarily to the applied strain and strain rate, and strain capacity and strain hardening susceptibility of the material. In confined systems, for example substrate supported polymer thin films, it may be expected that the presence of the underlying substrate affects the stress-strain behavior of the polymer film. On silicon supported PMMA thin films (120nm), Randall et al. noted that the tendency for pile-up during indentation is much greater than it is in the bulk material because the capacity for elastic-plastic deformation within the PMMA is severely constrained by the rigid substrate. This phenomenon will hereafter be referred to as strain shielding. In addition, the rheological anisotropy of interfacially confined thin films (see interfacial confinement) is expected to result in an inhomogeneous stress distribution, which will affect the thin film response to indentations.

References
18 N. P. Petrov, Friction in Machines and the Effect of the Lubricant, St. Petersburg, 1883).