Analytical method for the determination of indenter constants used in the analysis of nanoindentation loading curves

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We present a simple analytical method for the determination of indenter constants, key parameters used in the computation of the elastic modulus (or hardness) from the nanoindentation loading curve. In both forward and reverse analyses of the loading curve the proposed set of values are found to be more effective than those obtained empirically for the materials considered herein. Further improvement is anticipated as the accuracy of these constants depends on how precisely mechanical properties and curve fitting parameters are computed.

Keywords: Nanoindentation; Loading curve; Hardness; Elastic modulus

Analysis of nanoindentation data is carried out in two ways: (1) reverse analysis, where mechanical properties are extracted from the information contained in the load–displacement curves; (2) forward analysis, where the load–displacement curve is modeled from the known values of mechanical properties [1]. While reverse analysis is largely empirical, forward analysis is usually performed using the finite element method. A closed form solution appropriate for such analyses is difficult to develop because both stresses and displacements in the vicinity of the contact are non-uniform [2,3]. However, attempts are being made to come up with solutions that are based on theoretical underpinnings [4–6]. Hainsworth et al. [4] derived an expression capable of modeling a nanoindentation loading curve by relating the elastic and plastic deformations that occur during indentation with the characteristic contact radius of the indenter employed. The strength of this method is that it can be used for both reverse as well as forward analyses. For forward analysis, it offers an attractive alternative to the computationally expensive finite element method. However, the accuracy of the method hinges on the precise determination of indenter related constants as defined in Hainsworth et al. [4]. The basis of their determination was to obtain a best fit to experimental nanoindentation loading curves related to a wide range of materials having known mechanical properties with an assumed set of values for indenter constants. This empirical determination is cumbersome and perhaps the most perplexing aspect of the analysis of nanoindentation loading curves. Apart from this shortcoming, the method is only applicable to the case where the load–displacement relation during loading is parabolic. This relation holds when an ideally sharp conical indenter is employed in the indentation process. However, in reality the load–displacement relation is not parabolic due to bluntness in the indenter tip when the applied load is relatively small [4,6,7], the threshold being at 1 mN [6]. On the other hand, this method assumes that the mechanical properties are free from the indentation size effect which is normally obtained at higher loads. In other words, the method is only applicable for indentation depths where the effect of bluntness in the tip of the indenter and indentation size can be neglected. As the present study focuses on the development of a method for the determination of indenter constants, the above mentioned limitations apply here as well. The objectives of this study were to: (i) develop a method for the precise determination of indenter constants; (ii) check the effectiveness of the proposed set of values in modeling the loading curve and in determining the mechanical properties; (iii) clarify the misunder-
standing that a perfect fit guarantees precise determination of the mechanical properties.

Experiments have shown that indentation loading curves obtained with ideally sharp conical indenters (e.g. the Berkovich indenter) are usually well described by a linear relationship between applied load and displacement squared:

\[ P = K_{\text{exp}} \delta^2 \]

(1)

The proportionality constant \( K_{\text{exp}} \) is obtained through curve fitting and depends on the material as well as the type of indenter. By decomposing the total deformations that occur during indentation into its elastic and plastic components, Hainsworth et al. [4] derived a relationship which has the following form:

\[ P = K_m \delta^2 \]

(2)

\[ K_m = E \left( \frac{\phi_m}{H} + \frac{\psi_m}{E} \right)^{-2} \]

where \( E \) and \( H \) describe the modulus of elasticity and hardness of the material under consideration, respectively. Indenter constants \( \phi_m \) and \( \psi_m \) relate the characteristic contact radius of a rigid plastic indenter to the plastic and elastic components of total deformation, respectively. According to Hainsworth et al. [4] these constants depend only on the type of indenter used and are found to have the values 0.194 and 0.930, respectively, for a Berkovich indenter. The elastic modulus is then determined by equating \( K_m \) with the experimental \( K_{\text{exp}} \) from the known values of hardness and vice versa:

\[ E \left( \frac{\phi_m}{H} + \frac{\psi_m}{E} \right)^{-2} = K_{\text{exp}} \]

(3)

The essence of the application of Eq. (3) is that the experimental loading curve is fitted for successively assumed values of elastic modulus with known indenter constants and hardness of a material. A value that gives the best fit is then selected. Scrutiny of the nanoindentation data available in the literature [3,4], however, revealed that the prescribed values of the indenter constants yield spurious values of elastic modulus if the condition of perfect fit is enforced. We found several other pairs of indenter constants that can provide the same level of fit to the loading curve while maintaining constant values for the other two parameters in Eq. (3). This prompted the authors to devise a mathematical derivation with the aid of Eq. (3) that could address the significance of multiple values of indenter constants in fitting a loading curve. The subscript \( m \) is dropped from \( \phi_m \) and \( \psi_m \) in order to treat them as variables so that a relationship between the two can be established. Rearranging Eq. (3) in terms of \( \phi \) and \( \psi \) we have:

\[ \frac{\phi}{H^*} + \frac{\psi}{E^*} = 1 \]

in which

\[ H^* = \sqrt{\frac{P}{K_{\text{exp}}}} \quad E^* = \frac{E}{\sqrt{HK_{\text{exp}}}} \]

(5)

Eq. (4) represents a straight line with intercepts \( H^* \) and \( E^* \) in \( \phi \) and \( \psi \) space, respectively, as shown in Figure 1a, and provided the basis for the determination of indenter constants. Here, we define \( H^* \) and \( E^* \) as the normalized hardness and elastic modulus, respectively. It can be inferred that \( \phi \) and \( \psi \) are linearly dependent and any pair of values lying on the locus defined by Eq. (4) will give us the best fit to the experimental loading curve. While \( H \) and \( E \) are material specific, \( K_{\text{exp}} \) depends on both material type and the geometry of the indenter. Thus, the line represented by Eq. (4) is unique for a material and is obtained with the help of a sharp indenter having a specific geometry. Since proportionality constants depend only on the indenter geometry, all such lines obtained by indentation on different materials must intersect, as shown in Figure 1b. The coordinates of these intersecting points provide the required values of indenter constants for a given indenter geometry. Knowing \( \phi_m \) and \( \psi_m \), the normalized elastic modulus may be obtained by evaluating Eq. (4) at the point of intersection as:

\[ E^* = \frac{H^* \psi_m}{H^* - \phi_m} \]

(6)

Here as \( H^* \to \phi_m \), \( E^* \to \infty \), implying that the indenter constant \( \phi_m \) is the lowest value of normalized hardness. Thus, the normalized hardness determined for different materials must fall to the right side of \( \phi_m \), as shown in Figure 1b. Any values falling to the left of \( \phi_m \) must be ignored. A similar explanation can be furnished when normalized hardness is expressed as a function of indenter constants and normalized elastic modulus. Substituting the values of \( H^* \) and \( E^* \) into Eq. (6) gives:

\[ E = \frac{H \psi_m \sqrt{K_{\text{exp}}}}{\sqrt{H - \phi_m \sqrt{K_{\text{exp}}}}} \]

(7)

The method described in the previous section was verified with the experimental data scaled from Oliver and Pharr [3], in which a detailed description of the test method and materials are presented. Load–displacement data were acquired with a Berkovich indenter on aluminum, soda lime glass, sapphire, fused silica and tungsten. All of these materials were subjected to a maximum load of approximately 118 mN. The elastic modulus and hardness values given in Oliver and Pharr [3] were derived from the unloading curve using the Oliver–Pharr method and, for the sake of convenience, are displayed in Table 1. \( K_{\text{exp}} \) was determined using curve fitting techniques for all the materials considered here.

Figure 1. (a) Relationship between \( \phi \) and \( \psi \) and (b) family of lines in the \( \phi \) and \( \psi \) space.
and in all cases the coefficient of correlation was found to be very close to 1. For each of these materials the relationships between the indenter constants given by Eq. (4) are plotted in Figure 2. Except for the case of sapphire, all lines intersect at approximately $U_m = 0.182$ and $W_m = 0.725$, and are the required values of the constants for a Berkovich indenter. These values of indenter constants are close to the values obtained by Hainsworth et al., which are also shown in Figure 2. The fact that the line representing sapphire does not pass through the intersection point could be attributed to variability in the mechanical properties that exists within a material [8,9].

The apparent positions of lines in $U$ and $W$ space is also affected by the accuracy with which the fitting parameter $K_{exp}$ is calculated. It should be noted here that the fitting parameters largely depends on the number and positions of the data points in $P-h$ space and, therefore, can vary greatly, even within a material. Gong et al. [9] also reported that microstructural inhomogeneity has important implications for material responses to nanoindentation, which results in a large variation in the fitting parameters for the unloading curve. It can thus be

<table>
<thead>
<tr>
<th>Material</th>
<th>$E$ (GPa)</th>
<th>$H$ (GPa)</th>
<th>$K_{exp}$ (GPa)</th>
<th>Backcalculated, $E_{bc}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>70.4</td>
<td>0.21</td>
<td>5.81</td>
<td>18.76, -50.30</td>
</tr>
<tr>
<td>Soda lime glass</td>
<td>70.0</td>
<td>5.90</td>
<td>98.10</td>
<td>67.64, 107.08</td>
</tr>
<tr>
<td>Sapphire</td>
<td>403.0</td>
<td>26.90</td>
<td>442.00</td>
<td>301.44, 474.73</td>
</tr>
<tr>
<td>Fused silica</td>
<td>72.0</td>
<td>8.40</td>
<td>111.00</td>
<td>65.42, 96.33</td>
</tr>
<tr>
<td>Tungsten</td>
<td>409.8</td>
<td>3.80</td>
<td>107.00</td>
<td>427.02, -636.94</td>
</tr>
</tbody>
</table>

(1) This study; (2) Hainsworth et al.

![Figure 2. Determination of the indenter constant.](image)

![Figure 3. Comparison of theoretical predictions for loading curves with the experimental data.](image)
concluded that statistical analysis must be performed in order to obtain representative values of $E$, $H$ and $K_{\text{exp}}$ for a material to be used in the determination of the indenter constants.

Figure 3 shows a comparison of nanoindentation loading curves obtained theoretically using both sets of indenter constants with that obtained experimentally for all the materials considered here. The values suggested by Hainsworth et al. give a better approximation to the experimental curve in the cases of aluminum and sapphire because, as shown in Figure 2, the point (0.194, 0.930) lies closer to their lines, yet it does not give a best fit. The inability of Eq. (2) to model the experimental loading curve with $\Phi_m = 0.194$ and $\Psi_m = 0.930$ has also been reported in Kusano and Hutchings [10] and Pelletier [11]. However, it has also been reported that this pair yields satisfactory results in the case of coatings and thin films [4,12]. We are of the opinion that the best fit obtained using $\Phi_m = 0.194$ and $\Psi_m = 0.930$ does not mean that they are representative of indenter constants. Rather, this occurs due to the fact that the pair happens to lie on the line representing the material for which the best fit was obtained.

The effect of the level of fit on the back-calculated values of the elastic modulus (or hardness) requires further comment. The back-calculated elastic moduli are obtained using Eq. (7) for both sets of indenter constants, and are presented in Table 1. The back-calculated modulus $E_{\text{bc}}$ using $\Phi_m = 0.182$ and $\Psi_m = 0.725$ closely matches the actual elastic modulus $E$ for all those materials where the relationship between the load applied and the square of the penetration depth is perfectly linear and, at the same time, the corresponding material line in $\Phi$ and $\Psi$ space passes through the point of intersection. This is not always the case when $\Phi_m = 0.194$ and $\Psi_m = 0.930$. For aluminum and tungsten $\Phi_m = 0.194$ and $\Psi_m = 0.930$ gave negative values for the back-calculated elastic modulus and, hence, are absurd, for the reason previously described. Even the proposed set of indenter constants results in an unrealistic value for the elastic modulus in the case of aluminum, despite the fact that a reasonable agreement with the experimental curve was obtained. This is because the condition $P \propto h^2$ does not hold in this case, which, therefore, introduces error into the computation of $K_{\text{exp}}$. It is for this reason that a good level of fit to the experimental curve does not guarantee a realistic value of elastic modulus. Hence, precise determination of indenter constants requires high quality data for material constants (representative values of the mechanical properties) as well as a consistent curve fitting parameter from a wide range of materials.

This paper describes an analytical method for the determination of indenter constants, which are used for the forward as well as reverse analysis of nanoindentation loading curves obtained with an ideally sharp indenter. The method essentially consists of drawing several lines pertaining to different materials in $\Phi$ and $\Psi$ space and establishing the point of intersection. The apparent position of the line depends on how precisely the mechanical properties and curve fitting parameters for a given material are computed, and thus may greatly affect the indenter constants. Spurious values for the mechanical properties would be obtained if inappropriate indenter constants were introduced into the computation. This study also attempted to clarify that a good fit to the experimental curve does not ensure accuracy in measurement of the mechanical properties.