The sensitivity of the $\beta$-material parameter and its determination based on optimization of error in the mechanical properties

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1. Introduction

In recent times, efforts have been continuously applied to improve and/or develop a procedure for analyzing nanoindentation data to calculate mechanical properties for a wide range of materials. Several methods exist in the literature that includes both improved versions $[1-6]$ of the conventional Oliver and Pharr method used for determination of mechanical properties. Primarily, it may be determined both by fitting several sets of experimental data relating two nanomechanical quantities and from the total energy dissipated during indentation. While the first method is very cumbersome, the second may yield erroneous value for some materials. Reexamination of the experimental data on three ceramics available in the literature reveals that this parameter is very sensitive. Even a small error in its approximation may lead to a substantial error in the computation of initial unloading stiffness, contact area, elastic modulus and hardness values in certain conditions, and therefore, it must be determined very precisely. In this paper, a method for the determination of the $\beta$-material parameter based on the optimization of error in mechanical properties is proposed. $\beta$ determined using the proposed technique not only ensures the goodness of fit to the experimental data but also satisfies the loop conditions more precisely.

Abstract

The $\beta$-material is an extremely useful parameter for the analysis of nanoindentation data as it correlates different nanomechanical quantities and greatly simplifies the conventional Oliver and Pharr method used for determination of mechanical properties. Primarily, it may be determined both by fitting several sets of experimental data relating two nanomechanical quantities and from the total energy dissipated during indentation. While the first method is very cumbersome, the second may yield erroneous value for some materials. Reexamination of the experimental data on three ceramics available in the literature reveals that this parameter is very sensitive. Even a small error in its approximation may lead to a substantial error in the computation of initial unloading stiffness, contact area, elastic modulus and hardness values in certain conditions, and therefore, it must be determined very precisely. In this paper, a method for the determination of the $\beta$-material parameter based on the optimization of error in mechanical properties is proposed. $\beta$ determined using the proposed technique not only ensures the goodness of fit to the experimental data but also satisfies the loop conditions more precisely.

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Expressions represented by unified correlations diagram have one thing in common: the exponent of all these equations may be expressed as a fraction of a single quantity called $\beta$-material or simply $\beta$. This is an intrinsic material property that does not depend on the indentation size and can be directly used in determining various nanomechanical properties such as elastic modulus, hardness, yield strength, strain hardening exponent and fracture toughness of a material $[14]$. It is normally determined by fitting the experimental data between any two quantities in which one varies as the other whose exponent is a function of $\beta$. For example, $\beta$ can be determined by fitting the experimental maximum penetration depth ($h_{\text{max}}$) vs. peak indentation load ($F_{\text{max}}$) data in the form of $h_{\text{max}} = K_{\beta}^* F_{\text{max}}^{\beta^*}$. As the unified correlations diagram repre-
sents many such empirical equations, it would be cumbersome to acquire a unique value of $\beta$ that gives a perfect fit to all sets of the experimental data. This intrinsic material property may also be approximated using the total energy dissipated during loading [6,14]. As will be seen, $\beta$ determined using these two methods may differ remarkably and induces large error in the determination of nanomechanical quantities such as the initial unloading stiffness and area of contact, if employed incorrectly. It should be noted here that the $\beta$ value determined by either methods is greatly affected by the presence of residual stress [15,16]. The effect of residual stress is manifested when the two loading curves from the same material do not coincide or have different curvature. For instant, due to tensile residual stress, the resulting loading curve would appear below the actual loading curve in the load-displace- 
ment diagram thereby giving lower value of total work done (or a higher value of the total energy constant) and thus $\beta$ will be under 
estimated if second method is employed. The case is exactly oppo- 
site when the residual stress is compressive. Again, $\beta$ determined using the total energy constant may not describe the correlations between different nanomechanical quantities precisely as their relationship is only approximate. These shortcomings in the exist-
ing methods necessitate the development of a more rational ap-
proach for the determination of $\beta$. Thus, the objectives of this study are to: (i) examine the sensitivity of $\beta$ in the determination of various proportionality constants and nanomechanical quantities/properties in a systematic and quantitative manner; and (ii) propose a method for the precise determination of this useful material parameter based on the notion of error optimization so that the accuracy in the nanomechanical quantities/properties could be ensured.

2. Experimental data

This study uses the same set of experimental nanoindentation data given in Ref. [12], which were obtained from three ceramics viz. SiO$_2$, TiO$_2$ and Ta$_2$O$_5$ using Berkovich indenter subject to different peak indentation loads. These are elasto-plastic materials and show considerable elastic recovery (about 35%) upon unloading. Standard Oliver and Pharr method is applied to determine the initial unloading stiffness, contact area, and mechanical properties (summarized in Table 1) for these materials.

Fig. 1. (a) Typical nanoindentation load–displacement curve showing various nanomechanical quantities; (b) Unified correlations diagram showing relationships among several nanomechanical quantities [12]. Examples of loops: loop 1: $F_{\text{max}}$–$h_{\text{f}}$–$A_{\text{f}}$–$F_{\text{max}}$, loop 7: $F_{\text{max}}$–$h_{\text{f}}$–$A_{\text{f}}$–$F_{\text{max}}$. loop 4: $A_{\text{f}}$–$W_{\text{T}}$–$h_{\text{f}}$. loops 2 and 3 are identical to loop 1. Similarly, loop 5 and loop 6 resemble loop 7. $h_{i}$ ($i = c, P, f, max$) respectively denote contact, plastic, residual and maximum depth of penetration. Likewise, $W_{i}$ ($j = S, T, E, P$) describe absolute, total, elastic and plastic energies dissipated during indentation respectively.
3. Error analysis

3.1. Determination of mechanical properties

Following Attaf [6], the elastic modulus \( E_r \) and hardness \( H \) of a material can be determined respectively using following relations:

\[
E_r = K_E F_{\text{max}}^{1-2\beta} \tag{1}
\]

\[
H = K_H F_{\text{max}}^{1-2\beta} \tag{2}
\]

where

\[
K_E = \frac{\sqrt{\pi}}{2} \frac{K_p}{\sqrt{k_{\text{at}} F_{\text{max}}^2 (K_p - 1)}} \tag{3}
\]

Symbols \( K_{\text{at}}, K_H, \) and \( k_{\text{at}}^{\text{max}} \) are proportionality constants appearing respectively in expressions like: \( A_c = k_{\text{at}} F_{\text{max}}^2 \), \( h_{\text{max}} = K_{\text{at}} h_{\text{pl}} \), and \( h_{\text{pl}} = k_{\text{at}}^{\text{max}} F_{\text{max}} \), where \( A_c, F_{\text{max}}, h_{\text{max}}, h_{\text{pl}}, \) and \( h_{\text{pl}} \) respectively describe the contact area, peak indentation load, maximum, and plastic depths. Note that Eq. (1) is obtained when aforementioned correlations are substituted in the fundamental equation relating initial unloading stiffness, contact area and reduced elastic modulus.

As mentioned earlier, the parameter \( \beta \) may be determined by fitting several experimental data set in the form specified in the unified correlations diagram. Alternatively, it may be approximated using total energy dissipated during loading by the following relation:

\[
\beta = \frac{1}{2} \nu_t - 1 \tag{4}
\]

where, \( \nu_t \) is the total energy constant and is defined as a ratio of the absolute energy to the total energy. The absolute energy is the maximum possible energy that can be dissipated during indentation of a material and is used as reference energy to define other energy based parameters as well [12]. Mathematically, it is calculated by determining the area under the line joining the origin and peak indentation load point in the load–displacement curve. Values for the proportionality constants, total energy constant and \( \beta \) obtained by curve fitting for all these ceramics are summarized in Table 1. Eq. (4) overestimates the value of \( \beta \) by about 3%, 17% and 5% respectively for SiO\(_2\), TiO\(_2\) and Ta\(_2\)O\(_5\) in comparison to curve fitting method. Since it may be possible to obtain a large error in \( \beta \) for a material, it is extremely important to examine the effect of this error on the computed values of mechanical properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>( K_r )</th>
<th>( K_{hr} )</th>
<th>( K_{p}^{\text{max}} )</th>
<th>( \beta )</th>
<th>( \nu_t )</th>
<th>( E_r ) (GPa)</th>
<th>( H ) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO(_2)</td>
<td>1.7750</td>
<td>5.9872E–05</td>
<td>1.9440</td>
<td>0.5382</td>
<td>1.3985</td>
<td>71.9–67.7</td>
<td>9.24–8.32</td>
</tr>
<tr>
<td>TiO(_2)</td>
<td>1.4129</td>
<td>1.0344E–04</td>
<td>2.2196</td>
<td>0.5165</td>
<td>1.3263</td>
<td>113.5–99.5</td>
<td>7.73–7.21</td>
</tr>
<tr>
<td>Ta(_2)O(_5)</td>
<td>1.3780</td>
<td>6.9403E–05</td>
<td>1.7838</td>
<td>0.5535</td>
<td>1.3561</td>
<td>98.3–81.40</td>
<td>6.23–5.41</td>
</tr>
</tbody>
</table>

Fig. 2. (a) Error in the mechanical properties due to the change in \( \beta \) determined using Eq. (5); (b) shows that the error in the mechanical properties due to change in \( \beta \) is independent of the peak indentation load ; and (c) Error in other nanomechanical quantities due to change in \( \beta \) as given by Eq. (6).
associated with the error in $\beta$ may be analyzed based on how this particular parameter and other proportionality constants are determined.

3.2. When $\beta$ determined using total energy constant

First, let us assume that $\beta$ is calculated using Eq. (4) and proportionality factors are obtained by fitting the experimental data as shown in Table 1. Let $\beta_0$ and $\beta_1$ respectively denotes the exact and approximate values of $\beta$ for a given material. Then, the percentage error in the indentation modulus or hardness may be expressed in terms of $F_{\text{max}}$ and $\Delta \beta = \beta_0 - \beta_1$, as given by:

$$\Delta E, \text{ or } \Delta H = 1 - \frac{F_{\text{max}}^{\text{approx}}}{F_{\text{max}}^{\text{exact}}}$$

(5)

Fig. 2a shows the plot of the expression given by Eq. (5). From the figure, it is clear that even a small error in the value of $\beta$ introduces a significant error in the computed values of elastic modulus and hardness. For TiO$_2$, an error of 17% in $\beta$ would mean nearly 80% errors in both elastic modulus and hardness. This error is found to be independent of the peak indentation load as shown in Fig. 2b. Therefore, it may be concluded that Eq. (4) may not always yield good approximation for $\beta$. Further examination reveals that a similar variation could be seen if the same condition is employed in the evaluation of other quantities such as the initial unloading stiffness, contact area, work-of-indentation and maximum depth as a function of peak indentation load. In general, Eq. (5) may be written in the following form:

$$\Delta \Phi = 1 - \frac{F_{\text{max}}^{\text{approx}}}{F_{\text{max}}^{\text{exact}}}$$

(6)

where $n$ is an exponent that takes different values depending upon the two nanomechanical quantities involved in the correlation as shown in Fig. 2c. For instance, if Eq. (6) represents the relationship between the maximum depth of penetration and peak indentation load, the contact area and peak indentation load, the initial unloading stiffness and peak indentation load, the exponent $n$ takes the value of $-1$ to $1$, respectively. If the exponent is positive, an underestimation of $\beta$ would lead to a more serious error in the nanomechanical properties/quantities as can be seen in Fig. 2c. Conversely, an overestimation of $\beta$ has serious consequence if the exponent is negative.

3.3. When $\beta$ determined by curve fitting

In this case, we calculated the proportionality constants by enforcing several values of $\beta$ successively in the fitting process resulting in different values of $K_{\text{AF}}$ and $K_{\text{max}}^{\text{approx}}$ for almost same level of fit to the experimental data. Both are found to decrease exponentially with increasing values of $\beta$ as shown in Fig. 3a and b. If $K_{\text{AF}}$ and $K_{\text{max}}^{\text{approx}}$ correspond to a given $\beta$ used in the analysis, less error in the mechanical properties are obtained for all materials in this study as compared to the previous case. The error in the mechanical properties is particularly significant when the peak indentation load is comparatively small. At larger peak indentation load, the effect of changes in the proportionality constants and $\beta$ is negligibly small. In other words, several combinations of these parameters yield almost similar values of mechanical properties at large peak indentation load. But, $\beta$ and other proportionality constants incorporated in the unified correlations diagram are supposed to be unique for a given material. Thus, at this point, a question arises as which is the best combination for $\beta$ and proportionality constants so that the least error in the mechanical properties could be obtained for a given material. So far, only the goodness in the fit of the experimental curve is considered in the determination of these essential parameters. As mentioned, the unified correlations diagram comprises seven different loops and proportionality constants of each loop must satisfy certain mathematical conditions. For example, loop 1 encompasses all those relationship that exists between different depths of penetration and peak indentation load; peak indentation load and area of contact; and different depths of penetration and area of contact. By eliminating nanomechanical quantities involved in loop 1, a unique condition in terms of proportionality constants may be obtained. Following similar procedure, the loop conditions for all seven loops may be written respectively as:

$$\frac{K_{\text{AF}}^{\max}}{K_{\text{AF}}} = 1.0$$

(7)

$$\frac{K_{\text{AF}}}{\left(K_{\text{WF}}^{T} K_{\text{AF}}^{T}\right)^{\frac{1}{2}}} = 1.0$$

(8)

$$\frac{K_{\text{WF}}^{\max}}{\left(K_{\text{WF}}^{T} K_{\text{WF}}^{T}\right)^{\frac{1}{2}}} = 1.0$$

(9)

$$\frac{K_{\text{AF}}^{\max}}{\left(K_{\text{WF}}^{T} K_{\text{WF}}^{T}\right)^{\frac{1}{2}}} = 1.0$$

(10)

$$\frac{K_{\text{AF}}^{\max}}{\left(K_{\text{WF}}^{T} K_{\text{WF}}^{T}\right)^{\frac{1}{2}}} = 1.0$$

(11)
where \( K_{\text{max}}^{\text{Ah}} \), \( K_{\text{WF}}^{T} \), \( K_{\text{AW}}^{T} \), and \( K_{\text{SW}}^{T} \) respectively denote the proportionality constant between contact area and maximum penetration depth, total work done and peak indentation load, contact area and total work done, and maximum depth of penetration and total work done. There are as many as 33 such constants as shown in the unified correlations diagram. But only six of them are used to obtain above conditions relevant to each loop. The elastic modulus and hardness corresponding to each peak indentation load are calculated for all values of \( \beta \). While goodness in the fit is less affected, some of the loop conditions are easily perturbed by the change in \( \beta \) as shown in Table 2. Even a small perturbation (3% deviation) in the loop conditions has significant effect on the computed mechanical properties in some situations. For instance, an overestimation by 20% in \( \beta \) causes about a 3% deviation in loop conditions for loops 1, 4, 5 and 7 but induces nearly a 30% error in the mechanical properties for \( \text{SiO}_2 \) when the peak indentation load is 2448 \( \mu \text{N} \). Similar trend could be seen for other two materials as well. This is particularly important for materials such as thin films and coating where a restriction in maximum depth of penetration usually applies. However, at large peak indentation load, the error in the computed mechanical properties remain the same regardless of the error in \( \beta \) or deviation in loop conditions.

4. Proposed method to determine \( \beta \)

From the above discussions, it is clear that there must be compatibility among \( \beta \) and the proportionality constants in order to obtain precise value of the mechanical properties. This sort of compatibility could be ensured if, and only if, the values of these parameters obtained by curve fitting satisfy the loop conditions very precisely. But obtaining a best fit to several sets of experimental data with a unique value of \( \beta \) is a tedious task as several trial values are required to be selected. However, the fitting processes could be carried out with much ease if the error in the mechanical properties corresponding to each \( \beta \) is properly monitored.

In an attempt to facilitate the fitting process, the elastic modulus and hardness values for successive values of \( \beta \) are calculated using Eqs. (1)–(3) corresponding to each peak indentation load.
for all materials considered in this study. Note that the proportionality constants appearing in Eqs. (1)–(3) are obtained by fitting the respective experimental data corresponding to each value of $\beta$. Using the elastic modulus and hardness determined by the Oliver and Pharr method as reference values, the error associated with each of these values are evaluated and displayed in Fig. 4b and c for SiO$_2$, TiO$_2$, and Ta$_2$O$_5$. As mentioned earlier, the effect of change in $\beta$ is more pronounced at lower peak indentation load for all these materials. It is interesting to note that, in general, the errors in both elastic modulus and hardness are least for a particular value of $\beta$ irrespective of the peak indentation load. However, the shift in the lowest error point may be possible for a particular peak indentation load for some materials as shown in Fig. 4b and c. This discrepancy may be attributed to both relatively poor correlation between nanomechanical quantities involved in the computation and the computational error associated with reference values. Using error optimization technique, values of 0.5383, 0.5417 and 0.5533 for $\beta$ are obtained respectively for SiO$_2$, TiO$_2$, and Ta$_2$O$_5$ which are very close to the one reported in the literature except for TiO$_2$ [6]. For TiO$_2$, the value obtained for $\beta$ using the approach describe in this study is approximately 5% higher and 12% lower than that obtained respectively using curve fitting method and using total energy constant given by Eq. (4). This set of $\beta$ and proportionality constants gives the best fit to the experimental data and simultaneously satisfy all loop conditions precisely for all these materials.

As mentioned above, the mechanical properties obtained by the Oliver and Pharr method are used as reference values to determine $\beta$. But, expressions in the unified correlations diagram that do not involve $\beta$ may also be used to obtain the reference values for mechanical properties. For example, relations such as $A_c \propto h_{\text{max}}^2$ and $h_{\text{max}} \propto h_p$ may be used for this purpose. When these relationships are substituted in the fundamental relation, following equations for elastic modulus and hardness are obtained.

$$E_r = K_{EN} \frac{F_{\text{max}}}{h_p^2}$$  (14)

$$H = \frac{F_{\text{max}}}{K_{\text{Ah}} h_{\text{max}}}$$  (15)

where

$$K_{EN} = \sqrt{\frac{\pi}{4 K_p - 1}} \frac{1}{\sqrt{F_{\text{max}}}}$$  (16)

These equations yield accurate values of the elastic modulus and hardness as long as the relationships between the respective nano-mechanical quantities are perfectly linear. A comparison of the elastic modulus and hardness calculated using above equations is displayed in Fig. 5a and b, which is in excellent agreement with those obtained by the Oliver and Pharr method and using Eqs. (1)–(3). In this way, this parameter may be completely determined using equations contained in the unified correlations diagram and the correlation between maximum and plastic depth of penetration. Based on the above analysis, a step by step procedure for the determination of $\beta$ may be adopted as follows:

1. Determine the approximate value of $\beta$ from the total energy constant using Eq. (4).
2. Obtain reference values for the reduced elastic modulus and hardness using Eqs. (14)–(16) for all peak indentation loads.
3. Calculate the reduced elastic modulus and hardness using Eqs. (1)–(3) and determine the error associated with them using reference values obtained in step (2).
4. Repeat the above steps for several values of $\beta$.
5. Obtain a plot similar to the one shown in Fig. 4 and select the value of $\beta$ which gives the lowest error in both reduced elastic modulus and hardness for all peak indentation loads.

5. Conclusions

A sensitivity analysis of $\beta$ used in the determination of nanomechanical properties for a material is carried out in this study. As a result, it has been found that even a small error in it can significantly affect the accuracy of the mechanical properties in some conditions, if not employed appropriately. The situation becomes more severe when this parameter and other proportionality constants are calculated independently. If $\beta$ and other proportionality constants are determined by curve fitting, the effect of error in $\beta$ on mechanical properties becomes more pronounced only when the peak indentation load is relatively low for all the materials considered herein. To avoid uncertainty in its determination, a new method based on the optimization of error in the mechanical properties is proposed. The values thus obtained closely matches for SiO$_2$ and Ta$_2$O$_5$, but gives a higher value for TiO$_2$ as compared to those reported in the literature. The proposed method greatly simplifies its determination procedure, ensures the goodness in the fit to the various experimental data, and satisfies the loop conditions fairly accurately. However, this method requires that experimental data between the desired nanomechanical quantities be free from any residual stress effect.

Fig. 5. Comparison of: (a) elastic modulus and (b) hardness calculated by the Oliver and Pharr method ($E_r^\text{OP}$, $H^\text{OP}$) with those obtained using Eqs. (1)–(3) ($E_r$, $H$) and Eqs. (14)–(16) ($E_r$, $H$).
References