Influence of friction in material characterization in microindentation measurement

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Abstract

A comprehensive computational study is undertaken to identify the influence of friction in material characterization by indentation measurement based on elasto-plastic solids. The impacts of friction on load versus indentation depth curve, the values of calculated hardness and Young’s modulus in conical and spherical indentations are shown in this paper. The results clearly demonstrate that for some elasto-plastic materials, the curves of load versus indentation depth obtained either by spherical or conical indenters with different friction coefficients cannot be distinguished. However, if utilizing the parameter \( \beta \) (see text for details), to quantify the deformation of piling-up or sinking-in, it is easy to find that the influence of friction on piling-up or sinking-in in indentation is significant. Therefore, the material parameters which are related to the projected area will also have a large error caused by the influence of friction. The maximum differences on hardness and Young’s modulus can reach 14.59\% and 6.78\% respectively for some elastic materials shown in this paper. These results do not agree with those from researchers who stated that the instrumented indentation experiments are not significantly affected by friction.

Key words: Indentation; Friction; Piling-up or Sinking-in; Hardness; Finite element

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1 Introduction

The mechanical characterization of materials has long been represented by their hardness and Young’s modulus. In order to correctly estimate the hardness and Young’s modulus, depth-sensing indentation testing has been widely used as one of the advanced techniques in recent years. An advanced indentation instrument has the ability to continuously register the load $P$ versus the indentation depth $h$, during loading and unloading, and enables elastic and plastic mechanical properties of the indented materials to be evaluated [1, 2]. Particularly, it is possible to carry out this testing in microstructural scales (even in micro- and nanoscales), which makes this technique one of the most powerful tools for characterization of bulk and thin film materials. Experimental investigations of indentation have been conducted on many materials to extract hardness and other mechanical properties such as Young’s modulus, residual stress etc. [1, 3, 4]. Concurrently, comprehensive theoretical and computational studies have emerged to elucidate the contact mechanics and deformation mechanisms in order to systematically extract material properties from the curves obtained from instrumented indentation. For example, the hardness and Young’s modulus can be obtained from the maximum load and the initial unloading slope using the methods suggested by Oliver [1] and Tabor [4]. The elastic and plastic properties can be computed through a procedure proposed by Swadener [5], and the residual stresses can be extracted by the method of Suresh [6].

In the indentation studies mentioned above, the use of the finite element method (FEM) is an important tool for obtaining deeper understanding of the indentation measurement even for thin coating material [7–10]. The authors such as Yan [7] and Solberg [8] utilize 2D analysis, Antunes [9] and Youn [10], utilize 3D analysis to simulate the indentation process. However, in most of these indentation studies, for simplicity, no friction between the interfaces of indenter and specimen is taken into account. Furthermore, in recent published papers [6, 11, 12], FEM is employed extensively to study the stress fields in contact problems, as well as to predict the hardness and the development of surface deformation effects in indentation experiments. In those analyses, the authors assume that the friction has an insignificant effect in indentation. Nevertheless, in an indentation measurement with any kind of indenter (spherical, conical or Vickers etc.), the influence of friction in the contact area has been set forth [13–15]. Early in 1985, Johnson et al [14] first studied the influence of friction in indentation by recourse to the theory of the slip-line field. Such early investigations already indicated that an increase of up to 20% in hardness occurs for adhesive contacts as compared to frictionless ones. Besides this, according to the research of Hernot [15], if for the determination of...
Young’s modulus, piling-up or sinking-in is not taken into account, the error can reach 20%. More significantly, Mata [13] showed that the values of yield stress and work-hardening exponent, if extracted from the curves neglecting friction, may be up to 50% larger than the actual ones.

Although the aforementioned studies underline the characteristic features of frictional contact, a theoretical background to evaluate the influence of the friction on indentation measurement is still difficult to our knowledge. This numerical study intends to contribute to a deeper understanding of the influence of the friction coefficient in indentation. The comparisons on the values of calculated hardness and Young’s modulus are carried out both for the frictional and frictionless cases.

2 Theoretical and computational considerations

The typical $P - h$ curve response of an elasto-plastic material to sharp indentation is shown in Fig. 1. During loading, the response generally follows the relation described by Kick’s Law [3],

$$ P = Ch^2, $$

$$ C = H\alpha f, $$  

(1)

(2)

where $C$ is the loading curvature. $h$ is the penetration depth which can be directly measured by instrumented indentation. $H$ is the hardness of the material. $\alpha$ is a parameter that evaluates the piling-up or sinking-in of the material at the contact boundary, and $f$ is a geometrical factor. For conical indenters, $f = \pi\tan^2\theta$ with the half apex angle of conical indenter, $\theta$. Thus, for a typical $\theta = 70.3^\circ$, $f$ is equal to 24.504.

By recording data of the whole indentation procedure, the indentation hardness $H$ and Young’s modulus $E$ can be calculated as suggested by Oliver [1],

$$ H = \frac{P}{A_{\text{proj}}}, $$

$$ \frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}, $$  

(3)

(4)

where $A_{\text{proj}}$ is the projected contact area as shown in Fig. 2. $E_r$ is the so-called reduced modulus, which includes the material parameters of the indenter ($E_i, \nu_i$) and of the investigated material ($E, \nu$). Usually, the reduced modulus can be written as below:
where \( S = \frac{dP}{dh} \) is the initial unloading stiffness as shown in Fig. 1. The constant \( \gamma \) is relative to the indenters. If the indenter is conical, Berkovich or Vickers, \( \gamma \) equals 1.00, 1.034 or 1.012, respectively. Generally, the indenter is assumed as rigid \([1, 4]\), therefore, \( E_i \to \infty \). In Eq. (4), the last term, \((1-\nu_i^2)/E_i\) tends to zero. Then, Eq. (4) can be written into another form,

\[
E = (1 - \nu^2)E_r = \frac{(1 - \nu^2)\sqrt{\pi}}{2\gamma} \frac{S}{\sqrt{A_{proj}}}.
\]

According to Eqs. (1) and (2), Eq. (3) can be written as

\[
A_{proj} = \frac{P}{H} = \alpha f h^2.
\]

Considering piling-up or sinking-in, the projected contact area \( A_{proj} \) should be written as

\[
A_{proj} = f h_c^2,
\]

where \( h_c \) is the contact depth which incorporates piling-up or sinking-in as shown in Fig. 2. Equations (7) and (8) indicate that

\[
\sqrt{\alpha} = h_c/h.
\]
Therefore, if $\sqrt{\alpha} > 1$, piling-up occurs. On the other hand, $\sqrt{\alpha} < 1$ denotes sinking-in. In order to quantify the deformation of piling-up or sinking-in, according to Eq. (9), a derived parameter $\beta$ is introduced. It is defined as

$$\beta = \frac{h_c - h}{h} \times 100\% = (\sqrt{\alpha} - 1) \times 100\%.$$  \hspace{1cm} (10)

### 3 Numerical simulations

#### 3.1 The materials

The materials used in the numerical simulations are listed in Table. 1. For these elasto-plastic models, the material Young’s modulus is represented as $E$ and the initial yield stress is represented as $\sigma_y$. In general, the plastic behaviors of engineering metals can be closely approximated by the power law description [3],

$$\sigma = \begin{cases} 
    E \varepsilon & \text{for } \sigma \leq \sigma_y \\
    \frac{E^n}{\sigma_y^n} \varepsilon^n & \text{for } \sigma \geq \sigma_y 
\end{cases},$$  \hspace{1cm} (11)

where $\sigma$ and $\varepsilon$ is the true stress and strain, respectively. $n$ is the work-hardening exponent. In the following numerical calculations, the Poisson’s ratio is designated by $\nu$, and von Mises plasticity with $J_2$ flow theory is assumed. With the above assumptions and definitions, four independent parameters ($E$, $\nu$, $\sigma_y$, $n$) are required to completely characterize the elasto-plastic properties of the tested materials (see Table. 1).

<table>
<thead>
<tr>
<th>Name</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAF 2507 stainless steel[13]</td>
<td>$(E, \nu, \sigma_y, n)=$(200 GPa, 0.3, 675 MPa, 0.19)</td>
</tr>
<tr>
<td>Annealed copper[13]</td>
<td>$(E, \nu, \sigma_y, n)=$(110 GPa, 0.32, 20 MPa, 0.52)</td>
</tr>
<tr>
<td>Aluminum alloy[16]</td>
<td>$(E, \nu, \sigma_y, n)=$(70 GPa, 0.3, 500 MPa, 0.122)</td>
</tr>
<tr>
<td>Friction coefficient</td>
<td>$\mu$=0.0-1.0</td>
</tr>
</tbody>
</table>

#### 3.2 The computational models

A systematic numerical study is carried out with a variety of indenter geometries and friction coefficients. All numerical simulations are performed using the finite element code Metafor [17]. 2D axisymmetric finite element models are constructed to simulate the indentation response of elasto-plastic solids.
The finite element model with a conical indenter is shown in Fig. 3. In order to ensure the numerical accuracy, a finer mesh near the contact region and a gradually coarser mesh further away from the contact region are designed. The size of the specimen is $600 \mu m \times 600 \mu m$ and the finite element model is modeled using 1849 four-node quadrilateral elements, see Fig. 3. At maximum load, the minimum number of nodes in contact is never less than 15 in each FEM computation. $\theta$ is the half apex angle of the indenter, which is set to 63.14°, 70.3°, 75.79°, 80° and 81.5°, respectively, in this paper. And the maximum penetration depth is set to $h_{\text{max}} = 19.35 \mu m$ for all simulations with conical indenters.

Concurrently, the finite element model meshed for spherical indentation is similar to that shown in Fig. 3. But, it is modeled using 2500 four-node quadrilateral elements. The radius of the spherical indenter is represented by $R$, which is chosen as 1.25 mm and 0.25 mm, respectively. The size of the specimen is $8.0 mm \times 8.0 mm$ and $1.6 mm \times 1.6 mm$ for two indenters, respectively. According to Kucharski [12], with the radius of contact area, $a$, defined in Fig. 2, an indentation is shallow, if $a/R < 0.04$. Contrarily, $a/R > 0.04$, it is a deep indentation. In a shallow indentation, the calculation results are significantly influenced by the indenter size. In this paper, focusing on the influences of friction, deep indentation is chosen to make the calculation results insensitive to the size of the spherical indenter. Therefore, in the following FEM computations, for the spherical indenters with radii $R = 1.25 mm$ and $0.25 mm$, the maximum penetration depth is set to $h_{\text{max}} = 0.15 mm$ and 0.03 mm, respectively. At maximum load, the number of nodes in contact is never less than
26 in each FEM computation with different radii.

Measured friction coefficients show that the value of $\mu$ between well polished metallic surface and diamond lies within 0.1 to 0.15 [13]. In this paper, a wider range of $\mu$, which varies in the range of 0.0-1.0, is adopted for all the simulations with spherical and conical indenters.

4 Results and discussions

4.1 Computational results and comparison for conical indenters

The $P - h$ curves obtained for SAF 2507 stainless steel, annealed copper and aluminum alloy with different friction coefficients are shown in Fig. 4(a) to Fig. 6(a). Although the friction coefficient $\mu$ varies from 0.0 to 1.0, the $P - h$ curves obtained for a given half apex angle are nearly identical. The maximum difference values of $P_{\text{max}}$ in Fig. 4(a) to Fig. 6(a) are all lower than 3.02%. However, this does not mean that there is no visible impact on the calculated hardness and Young’s modulus, because they have a direct correlation with the projected contact area $A_{\text{proj}}$, and $A_{\text{proj}}$ is a function of $h_c$ which is related to piling-up or sinking-in. The values of piling-up or sinking-in obtained in different friction cases may be absolutely different.

![Fig. 4. Calculation results as obtained with conical indenter for SAF 2507.](image)

(a) $P - h$ curves for varying $\mu$  
(b) $\beta$ versus $\theta$ for varying $\mu$

Therefore, in a further study, the relationships between $\beta$ and the friction coefficient $\mu$ for different conical indenters are shown in Fig. 4(b) to Fig. 6(b). For all materials, the values of $\beta$ tend to decrease with an increase of $\mu$. The curve for a larger $\mu$ is always below the curve for a smaller $\mu$. This means adopting a larger $\mu$ can effectively restrain piling-up from growing, especially with a smaller half apex angle. For example, in Fig. 4(b), in the
case of $\theta = 70.3^\circ$, while $\mu < 0.1$, the values of $\beta$ are above zero, the material around indenter is piling-up. But following an increase of $\mu$, $\beta$ becomes smaller, piling-up disappears and sinking-in appears. Besides this, in Fig. 4(b), we can see that with the same value of $\mu$, when the half apex angle increases, the amount of piling-up decreases, and sinking-in tends to occur. For $\mu = 0$ and $\theta = 63.14^\circ$, piling-up takes place, but when $\theta = 81.5^\circ$, sinking-in occurs. This means that in the indentation measurement with a smaller half apex angle, piling-up will be favored. On the other hand, when the half apex angle is large enough, piling-up may be replaced by sinking-in. The foregoing phenomena also appear in Fig. 5(b), for annealed copper, and in Fig. 6(b), for aluminum alloy. Here, we should note that in Fig. 5(b), the values of $\beta$ are always below zero, which denotes that only sinking-in occurs for annealed copper, as it has a larger work-hardening exponent $n$ and a smaller ratio $\sigma_y/E$. This is in good agreement with the results presented in Alcalá et al [18].

Furthermore, for every material, it is clear to see, in the case of $\theta = 63.14^\circ$, the differences on the deformations of piling-up or sinking-in with different
\(\mu (\mu < 0.3)\) are obvious and with an increase of \(\theta\), the differences decrease. Concurrently, it is noted that when \(\mu > 0.3\), \(\beta\) remains unchanged following an increase in \(\mu\). Investigating the deformed mesh, we find that the nodes on the interfaces of indenter and specimen remain sticking, which leads to nearly the same values of piling-up or sinking-in although the friction coefficients are different. However, this also strongly depends on the half apex angle \(\theta\). When \(\theta\) is larger, the value of \(\beta\) becomes nearly independent of the friction coefficient \(\mu\). Such as in Fig. 4(b), in the cases of \(\theta \geq 80^\circ\), these differences are invisible. The nodes on the interfaces are found to have nearly no slip when studying their displacements in tangent direction. Those phenomena denote that the value of \(\beta\) is less affected by friction coefficient if the indentation is performed using a conical indenter with a larger half apex angle.

4.2 Computational results and comparison for spherical indenters

![Graphs for spherical indenters](image-url)

Fig. 7. Numerical \(P - h\) curves as obtained with spherical indenter for SAF 2507.

![Graphs for spherical indenters](image-url)

Fig. 8. Numerical \(P - h\) curves as obtained with spherical indenter for annealed copper.
Fig. 9. Numerical $P - h$ curves as obtained with spherical indenter for aluminum alloy.

Fig. 10. The curves of $\beta$ versus friction coefficient $\mu$.

Fig. 7–Fig. 9 show the $P - h$ curves obtained by two spherical indenters with different friction coefficients for the three materials listed in Table 1. The
$P - h$ curves in Figs. 7(a), 8(a) and 9(a) are obtained using a bigger spherical indenter ($R = 1.25 \text{ mm}$). In Fig. 7(b), 8(b) and 9(b), the $P - h$ curves are obtained using a smaller spherical indenter ($R = 0.25 \text{ mm}$). Although the friction coefficient $\mu$ varies from 0.0 to 1.0, the $P - h$ curves can not be distinguished, which is similar to those obtained by conical indenters. The maximum differences in the values of $P_{\text{max}}$ are all lower than 1.38% for all materials. A larger friction coefficient can effectively constrain the piling-up or lead to an increase in the amount of sinking-in. However, when $\mu > 0.3$, the nodes on the interfaces are sticking and the amount of sinking-in tends to be constant. Moreover, the curves of $\beta$ versus $\mu$, obtained for two different spherical indenters are almost the same, see Fig. 10. This denotes that the effect of friction has less correlation with the radius of spherical indenter.

4.3 Discussions

From the foregoing comparisons, we can see that the $P - h$ curves obtained either by conical or spherical indenters with different friction coefficients show very little differences. However, through the parameter $\beta$, the influence of friction on piling-up or sinking-in is clearly highlighted. In Tables. 2 to 4, we illustrate explicitly how this impact reflects on the calculated hardness and Young’s modulus. The $H$ and $E$ remarked by “O&P” denotes that the contact depth $h_c$ is calculated by the classical method of Oliver and Pharr [1], which is written as bellow,

$$h_c = h_{\text{max}} - \epsilon \frac{P_{\text{max}}}{S}, \quad (12)$$

where the geometric constant, $\epsilon$ is defined as $\epsilon=0.72$ for conical indenter and $\epsilon=0.75$ for spherical indenter. For the $H$ and $E$ denoted by “FEM”, the contact depth, $h_c$ is directly determined in the foregoing FEM simulations.

The results obtained from FEM and the O&P methods tend to be significantly different. This is because the contact depth calculated by Eq.12 does not take into account the effect of friction on piling-up and sinking-in. According to Eq.12, $h_c$ is derived from the $P - h$ curve. Moreover, the $P - h$ curves are almost identical although the friction coefficient is varied in a large range. Thus, $h_c$ are nearly identical.

From the results obtained by FEM method, it can be clearly seen that the friction coefficient obviously affects the calculated values of hardness and Young’s modulus. The hardness has a tendency to increase and Young’s modulus tends to decrease with an increase of friction coefficient. When the $\mu > 0.3$, the hardness and Young’s modulus stay constant, particularly in the spherical simula-
tions. As for the coefficient $\beta$, it can be seen that above a friction coefficient of $\mu > 0.3$, the hardness and Young’s modulus tend to be constant because the material sticks to the indenter on the contact interface.

The maximum differences of hardness caused by friction coefficient reach 14.59% in the conical simulation for Aluminum alloy. And the maximum errors of Young’s modulus reach 6.78% in the spherical simulation for annealed copper.

Thus, we can say that from the foregoing analysis results, the errors of calculated hardness and Young’s modulus caused by the influence of friction are significant and should not be neglected.

Table. 2. SAF 2507 stainless steel.

<table>
<thead>
<tr>
<th>$\mu$</th>
<th>Conical indenter ($\theta = 63.14^\circ$)</th>
<th>Spherical indenter</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>$H$ (GPa)</td>
<td>$E$ (GPa)</td>
</tr>
<tr>
<td>0.0</td>
<td>4.22</td>
<td>3.54</td>
</tr>
<tr>
<td>0.05</td>
<td>4.27</td>
<td>3.71</td>
</tr>
<tr>
<td>0.1</td>
<td>4.33</td>
<td>3.82</td>
</tr>
<tr>
<td>0.15</td>
<td>4.34</td>
<td>3.91</td>
</tr>
<tr>
<td>0.3</td>
<td>4.31</td>
<td>3.98</td>
</tr>
<tr>
<td>0.6</td>
<td>4.31</td>
<td>3.98</td>
</tr>
<tr>
<td>1.0</td>
<td>4.37</td>
<td>4.03</td>
</tr>
</tbody>
</table>

Table. 3. Annealed copper.

<table>
<thead>
<tr>
<th>$\mu$</th>
<th>Conical indenter ($\theta = 63.14^\circ$)</th>
<th>Spherical indenter</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$H$ (GPa)</td>
<td>$E$ (GPa)</td>
</tr>
<tr>
<td>0.0</td>
<td>1.38</td>
<td>1.56</td>
</tr>
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<td>1.39</td>
<td>1.59</td>
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<tr>
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<td>1.39</td>
<td>1.60</td>
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</tr>
<tr>
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<td>1.65</td>
</tr>
<tr>
<td>0.6</td>
<td>1.40</td>
<td>1.65</td>
</tr>
<tr>
<td>1.0</td>
<td>1.40</td>
<td>1.65</td>
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Table. 4. Aluminum alloy.

<table>
<thead>
<tr>
<th>$\mu$</th>
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<th>Spherical indenter</th>
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<tr>
<td></td>
<td>$H$ (GPa)</td>
<td>$E$ (GPa)</td>
</tr>
<tr>
<td>0.0</td>
<td>2.26</td>
<td>1.85</td>
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<td>0.05</td>
<td>2.34</td>
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</tr>
<tr>
<td>0.1</td>
<td>2.32</td>
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</tr>
<tr>
<td>0.15</td>
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<td>2.09</td>
</tr>
<tr>
<td>1.0</td>
<td>2.29</td>
<td>2.09</td>
</tr>
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The influence of friction in indentation testing with conical and spherical indenters is studied in this paper. We find that for some elasto-plastic materials, the $P-h$ curves obtained either by spherical or conical indenters with different friction coefficients in the range of engineering metals can not be distinguished. Then, we introduce a parameter $\beta$ to evaluate piling-up or sinking-in. The figures 4(b), 5(b) and 6(b) clearly show the friction between indenter and specimen can significantly affect the the amount of piling-up or sinking-in in the simulation with conical indenter, which has a significant effect on the contact area. Especially when the half apex angle is smaller, the influence of friction is obvious. Friction can effectively impede the slip of material on the interfaces between indenter and specimen, which leads to a decrease in the amount of piling-up, or an increase of sinking-in. However, when the half apex angle is large enough, e.g. $\theta \geq 80^\circ$, friction becomes predominant, because the material on the interfaces between indenter and specimen easily tend to be adhered on indenter. In indentations with spherical indenters, the amount of piling-up decreases or sinking-in increases with an increase of friction coefficient while $\mu < 0.3$. Moreover, the effect of friction seems to be independent of the radius of the spherical indenter. As can be seen in Fig. 10, the curves of $\beta$ versus $\mu$ obtained by two different spherical indenters with varying friction coefficients do not have significant differences.

The friction between indenter and specimen can significantly affect the contact area and piling-up or sinking-in. Therefore, the values of hardness and Young’s modulus, which are related to the projected contact area $A_{proj}$, should be significantly different too. For some materials, the maximum differences of hardness and Young’s modulus can reach 14.59% and 6.78%, respectively, for friction and frictionless cases, which contradicts the assumption made by several researchers that the instrumented indentation experiments are not significantly affected by friction [6, 11, 12].

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