A dual conical indentation technique based on FEA solutions for property evaluation

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ABSTRACT

The sharp indenters such as Berkovich and conical indenters have a geometrical self-similarity so that we can obtain only one parameter from an indentation loading curve, which makes different materials have the same load–displacement relation. Most studies to evaluate elastic–plastic properties by using the geometrical self-similar indenter have therefore tried to use dual/plural indentation techniques, on the basis of the concept of representative strain/stress varying with the indenter angle. However, any suggested representative concept is not universally operative for real materials. In this work, we suggest a method of material property evaluation without using the concept of representative strain. We begin the work by studying the characteristics of load–depth curves of conical indenters via finite element (FE) method. From FE analyses of dual-conical indentation, we investigate the relationships between indentation parameters and load–depth curves. The projected contact diameter is expressed as a function of the indenter angle, tip-radius, and material properties, which allows us to simply predict the elastic modulus. Two mapping functions for two indenter angles (45° and 70.3°) are presented to find the two unknowns (yield strain and strain-hardening exponent) via dual indentation technique. The method provides elastic modulus, yield strength and strain-hardening exponent with an average error of less than 5%. The method is valid for any elastically deforming indenters. We also discuss the sensitivity of measured properties to the load–displacement curve variation, and the difference between conical and Berkovich indenters.

1. Introduction

The instrumented indentation test is a method to extract material properties from indentation load–depth curves with micro specimens or parts of mechanical structures in use (Chen et al., 2006, 2010; Cook and Pharr, 1990; Giannakopoulos and Larsson, 1997; Giannakopoulos and Suresh, 1999; Huber and Tsakmakis, 1999a,b; Kermouche et al., 2008; Lan and Venkatesh, 2007; Lee et al., 2005; Liao et al., 2009; Ogawara et al., 2006a,b, 2009; Oliver and Pharr, 1992; Suresh and Giannakopoulos, 1998; Xia et al., 2007). The contact area and load–displacement curves to measure the hardness and elastic modulus are fundamental and important data for indentation test. However, it is quite difficult to measure or predict the contact diameter due to imperfection or tip-blunting of indenter. Based on Hertz contact mechanics, many studies on evaluating the actual contact area and geometric deviation of indenter from its nominal geometry have been performed (Borodich et al., 2003; Borodich and Keer, 2004).

There should be a one-to-one match between the load–depth curve obtained from an indentation test and its material properties. However, some materials having different material properties may show the same load–depth curve due to the geometrical self-similarity of sharp indenters (Capehart and Cheng, 2003; Chen et al., 2007; Cheng and Cheng, 1998; Lee et al., 2008; Tho et al., 2004). Chen
et al. (2007) and Lee et al. (2008) demonstrated that countless materials can have the same Kick's law coefficient \( C \) despite their different material properties. Dual (plural) sharp indenters with dissimilar angles can be therefore used to solve this problem. In previous studies (Bucaille et al., 2003; Cao and Lu, 2004; Chollacoop et al., 2003; Ogasawara et al., 2005, 2006a; Shim et al., 2008), unique solutions were attempted by using the concept of representative strain \( \varepsilon_R \) which varies with the half-included angle of indenter.

The loading curves from sharp indenters generally follow the Kick's law relation.

\[
P = C h^2
\]

(1)

Here \( P \) is the indentation load, \( h \) is the measured depth from reference surface and \( C \) is the coefficient of the Kick's law. \( P_{\text{max}} \) is the maximum load at the maximum indentation depth \( h_{\text{max}} \), and the initial unloading slope \( S \) is defined as \( dP/dh \) at \( h = h_{\text{max}} \) as depicted in Fig. 1. Kick's law is valid for the ideal sharp indenter, but the tip-radius effect breaks the Kick's law and the corresponding self-similarity. For eliminating the effect of tip-radius, Lee et al. (2008) suggested the corrected Kick's law as following equation.

\[
P = C (h + h_g)^2, \quad h_g = R \left( \frac{1}{\sin \alpha} - 1 \right)
\]

(2)

Here \( h_g \) is the gap between the indenters with zero and finite tip-radius (Fig. 2). Eq. (2) is not valid when the effect of the tip-radius is dominant in shallow indentation. However, as the indentation depth increases the effect of the tip-radius decreases so Eq. (2) became valid. In Fig. 2, \( h_t \) means the expected indentation depth \( (h_t = h + h_g) \) for an ideally sharp indenter.
$P/h^2$ is a constant value independent of indentation depth, so the representative strain which shows the deformation state of materials has a unique value. In most previous studies, the main objective is to determine the way of defining the representative strain and its value. They tried to reduce the number of indentation parameters using the representative strain. The concept of the representative strain was introduced by Tabor (1951) ($e_R = 0.08$ for Vickers indentation). Dao et al. (2001) ($e_R = 0.033$) found out that stress–strain curves of materials having the same Kick’s law coefficient $C$ exhibit the same true stress at the plastic strain of 3.3% for $70.3^\circ$ conical indentation. Dao et al. (2001) presented then the following dimensionless function.

$$
\Pi\left(\frac{E_x}{\sigma_k}, n\right) = \frac{C}{\sigma_k}
$$

(3)

Here $\sigma_k$ is the representative stress corresponding to $e_k$, $n$ is the strain-hardening exponent, and $E_x$ is the effective modulus. Dao et al. (2001) used the following relation between representative stress and representative strain.

$$
\sigma_R = \sigma_o \left(1 + \frac{E}{\sigma_o e_k}\right)^n
$$

(4)

Here $\sigma_o$ is the yield strength and $E$ is the elastic modulus. The concepts of dual conical indentation were suggested by Bucaille et al. (2003) ($e_R = 0.105 \cot \alpha$) and Chollacoop et al. (2003) ($e_R = -2.185 \times 10^{-3} \alpha + 0.1894$). Here $\alpha$ is the half-included angle of a conical indenter (Fig. 2). They ob-

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Fig. 4. Load–depth curves for various values of indentation parameters.

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Fig. 5. $P/R_{\max}$ vs. $h^2$ curves for different material properties with the same $C$ and $E$ for (a) $\alpha = 70.3^\circ$ and (b) $\alpha = 45^\circ$. 
obtained multiple equations by substituting $e_R$ and $r$ values of two indenter angles into Eq. (4), and then determined $\sigma_o$ and $n$. Thereafter, Cao and Lu (2004) suggested some conditions which should be satisfied to use the above method. Ogasawara et al. (2005) ($e_R = 0.0139 \cot \alpha$ for $70.3^\circ$ conical indentation defined the representative strain as a function of plastic strain related to axisymmetric deformation. Their representative stress corresponding to the representative strain is determined by the following Eq. (5).

$$\sigma_R = R \left( 2 \frac{\sigma_k}{E} + 2n \right)^n$$

Chen et al. (2007) and Ogasawara et al. (2006) noted that $C/\sigma_k$ varies linearly with respect to $E/\sigma_k$ when its value is very small (elastic limit), and $C/\sigma_k$ asymptotically approaches to a constant value as $E/\sigma_k$ increases (rigid plastic limit). They suggested the function $C/\sigma_k$ using the elastic limit and the rigid plastic limit.

$$P = C = \left( \frac{1}{m_e E} + \frac{1}{m_p} \right)^{-1}$$

Here $m_e$ is the slope of the linear region, $m_p$ is the rigid plastic limit. However, some materials having different properties still show almost the same $C$ in dual (or plural) indentation, so Chen et al. (2007) named them “mystical materials”. As the difference of the angles, $\Delta \alpha$, gets bigger, the region of “mystical materials” decreases. However, Chen et al. (2007) have shown neither a systematic way

<table>
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<tr>
<th>$\alpha$ (°)</th>
<th>Kick's law coefficient $C$ (GPa)</th>
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<tbody>
<tr>
<td>$\sigma_o = 800$ MPa</td>
<td>$\sigma_o = 420$ MPa</td>
</tr>
<tr>
<td>$n = 5.0$</td>
<td>$n = 2.6$</td>
</tr>
</tbody>
</table>

| $\sigma_o = 800$ MPa | $\sigma_o = 600$ MPa |
| $n = 5.0$ | $n = 3.7$ |

| Gap (%) | 14.9 | 7.6 | 0.3 |
| Gap (%) | 6.4 | 4.3 | 4.5 |
| Gap (%) | 0.6 | 96.5 | 88.7 |

Table 1
Comparison of $C$ values with three half-included angles.

Table 2
Material properties for FE analyses.

<table>
<thead>
<tr>
<th>Material property of indenters</th>
<th>Material property</th>
<th>Values used in FEA</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC ($E_I = 537$ GPa, $v_I = 0.24$)</td>
<td>Young's modulus (GPa)</td>
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<td></td>
<td>Poisson's ratio</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>Yield strength (MPa)</td>
<td>200, 400, 600, 800, 1200, 1600, 2000</td>
</tr>
<tr>
<td></td>
<td>Strain-hardening exponent</td>
<td>1.1, 2, 3, 5, 7, 10, 13, 20</td>
</tr>
</tbody>
</table>

**Fig. 6.** $c^2$ vs. indentation depths for four different yield strains.

**Fig. 7.** $c^2$ vs. $1/n$ for three different various tip radii.

**Fig. 8.** $c^2$ vs. $1/n$ curves for various values of yield strain.
of distinction nor an application to the non-power law materials.

As was stated above, the definitions and the variations of the representative strain with indenter angles were different in each previous study. In addition, suggested definitions of $\varepsilon_R$ are disputable in their representativeness (Lee et al., 2010). We can imagine that two true stress–strain curves could be almost the same until the strain is less than $\varepsilon_R$ but be different out of the region. If the representative strain is a function of indenter angle only, though

![Fig. 9. 3D distribution of $C/E$ & contour lines (a) $\alpha = 45^\circ$, (b) $\alpha = 70.3^\circ$.](image1)

![Fig. 10. Deference of angles between gradients of each contour line.](image2)

![Fig. 11. $C/E$ vs. $1/n$ curves for various values of yield strain.](image3)
material properties are different, two materials must have the same \( \epsilon_R \) and \( \sigma_R \). If the representative strain is smaller than about 0.3, the load–depth curves of two materials are different. This means that two materials which show the different load–depth curves have same \( \epsilon_R \) and \( \sigma_R \). Therefore, the representative strain must have a large value. Chaudhri (1998) and Giannakopoulos and Suresh (1999) suggested the \( \epsilon_R = 0.25–0.36 \), but these values are too large to present the strain field of subindenter. To be representative, \( \epsilon_R \) and \( \sigma_R \) should therefore vary with the material properties, and be a unique value for each material.

Swaddiwudhipong et al. (2005) and Le (2008, 2009) found the relationship between the characteristics of the load–displacement curves and the properties of elastic–plastic materials. Then, they built a reverse analysis algorithm for dual indentation. They predicted material properties using the change of various indentation variables such as \( C \), unloading slope, and loading–unloading energies. Swaddiwudhipong et al. (2005) used the ratio of \( E/\sigma_o \) and \( n \) as two key parameters. However, the effective Young’s modulus \( E_e \) cannot express the real material property, and the ratio of the Young’s modulus to yield strength \( E/\sigma_o \) is more important than the ratio of the effective modulus to yield strength \( E_e/\sigma_o \) based on our analysis. In addition, the maximum variation of predicted yield strength with experimental scatter for the loading curve is about 70%.

Like these, prior indentation theories have inherent restrictions on measurement of practical material properties. Even though FE analysis was used in some works, limited range of material property was considered and inappropriate methods were used as mentioned above. Only with verification for a wider range of real materials, the previous methods could be practical and reliable.

In this work, we suggest a method of material property evaluation by using a reverse analysis, which does not adopt the concept of representative strain. In the spherical indentation, it is difficult to evaluate the material properties without the defining a representative strain. In contrast, when geometrically self-similar indenters are used, the multiple indentations can be used to obtain the equations as many as the unknown values and it does not require defining the representative strain value. To establish the equations, we investigate the aspect of material deformations (pile-up/sink-in) near the contact area. If the measured (or calculated) contact area is inaccurate, large errors in predicting the elastic modulus or hardness may accompany. We also examine the contact mechanics of indenter with tip radius. In engineering practice, the indenter is not perfectly sharp but has a tip radius. Therefore, in shallow indentation there is only spherical contact; the material deformation in deep indentation obeys the conical contact mechanics. In this work, we analyzed the variation of contact radius and load–displacement curves with indenter tip radius. Based on the method proposed by Lee et al. (2008), we removed the effect of tip radius. Moreover, we investigate the variation of load–displacement curve with indent angle.

The paper is organized as follows. Section 2 shows the FE model (ABAQUS, 2007) and conditions for conical indentation. We observe the effect of material properties, friction coefficient and conical indenter’s angle on the load–depth curves in Section 3. Section 4 presents the regression functions using the correlation among yield strains, strain hardening exponents and indentation load–depth curves. In addition, we provide the calculation method for contact diameter using regression function of material properties. We then propose a dual–conical indentation technique and verify the method. We also discuss the sensitivity of measured properties on the load–depth curve in Section 5. Finally, Section 6 shows the difference between conical and Berkovich indenters.

### 2. Finite element analysis

Fig. 3 shows the 2D FE model of the numerical conical indentation test that has the same projected contact area as the Berkovich indenter for the same indentation depth. We perform the finite element (FE) analyses using ABAQUS. We make an axisymmetric model considering that both load and configuration are axisymmetric. We carry out nonlinear geometry analyses using isotropic elastic–plastic material, which obeys \( J_2 \) flow theory.

We use the four-node axisymmetric element CAX4 (ABAQUS, 2007). We performed finite element analyses while changing the size of minimum element \( e \), according to the indenter tip-radius \( R \). It turned out that when \( R/e > 120 \), the element size effect is negligible, so we choose \( R/e = 160 \) as the minimum element size. Multi-Point Constraints (MPC) is conveniently used at the transition region where element size changes, but constrained mid-nodes of MPC tend to give discrete stress–strain values. We thus adopt trapezoidal elements in the transition region near the contact surface, and use MPC in only the transition region far from the contact surface. FE model consists of about 24900 4-node axisymmetric elements (CAX4) and 25400 nodes. We place contact surfaces at both material and indenter surfaces. Axisymmetric boundary conditions are imposed on the nodes on the axisymmetric axis. The indenter moves down to penetrate the material the bottom of which is fixed.

The Young’s modulus and Poisson’s ratio of WC indenter are taken by an ultrasonic test, which values are 537 GPa.
and 0.24, respectively. To confirm the effect of the tip-radii, we vary the ratio of the tip-radius to the maximum indentation depth $R/h_{\text{max}}$ between 0.5 and 2. Indentation formulas that developed WC indenter are valid even when Young’s modulus and Poisson’s ratio of WC indenter are substituted with those of diamond (Lee et al., 2008).

![Flow chart for determination of material properties](image)

Fig. 13. Flow chart for determination of material properties.
We need a regression equation for stress–strain curve and can be expressed with piecewise power relationship (Rice and Rosengren, 1968).

\[
\varepsilon_e = \begin{cases} 
\frac{\sigma}{E} & \text{for } \sigma \leq \sigma_y \\
\left(\frac{\sigma}{\sigma_y}\right)^n & \text{for } \sigma > \sigma_y, \quad 1 < n \leq \infty
\end{cases}
\]  

(7)

Here \(\varepsilon_y \equiv \sigma_y/E\) is the yield strain and \(n\) is the strain-hardening exponent. Total strain \(\varepsilon_e\) is decomposed into elastic and plastic strains \((\varepsilon_e = \varepsilon_y + \varepsilon_p)\). While many indentation studies generally used Ramberg–Osgood stress–strain relation, we in the present work adopt Eq. (7), since the latter provides a distinctive linear elastic region and consequently distinctive yield strength.

### 3. Characteristics of indentation deformation

#### 3.1. Deformation under indenter and characteristic of load–depth curves

We investigated the effects of friction coefficient, Young’s modulus, yield strength and strain hardening exponent on load–depth curves via indentation FE analyses. Fig. 4a shows load–depth curves with variation of friction coefficient. Here Coulomb friction coefficient \(f\) is varied from 0.1 to 0.4. Although load increases slightly with friction coefficient \(f\) for the same indentation depth, the difference is virtually negligible. While distributions of stress and strain are substantially affected by the friction coefficient, load–depth curves are almost independent of friction coefficient. In this study, a method for the evaluation of material properties is proposed with the value \(f=0.3\), and the sensitivity of the method on the friction coefficient is then discussed.

Fig. 4b shows the relations between modulus and load–depth curve. Indentation load increases for the same indentation depth as the Young’s modulus increases. The effect of Young’s modulus on load–depth curves is less than the other parameters but the effect cannot be ignored. It is noteworthy that unloading curves vary sensitively with Young’s modulus. Fig. 4c and d shows that indentation load increases with yield strength, but decreases with strain hardening exponent for the same indentation depth. In other words, all the material properties are related to loading curves, and the slope of unloading curves is closely related to Young’s modulus.

#### 3.2. Characteristic of load–depth curves with indenter angles

Lee et al. (2008) reported that there are countless materials which have the same Kick’s law coefficient \(C\) despite their different material properties. It means that stress–strain curves obtained from load–depth curves for self-similar indenters cannot be unique.

Fig. 5 shows the variations of load–displacement curves for two sets of arbitrary materials with three different half included angles of indenter, \(\alpha = 70.3^\circ\), 60.0\(^\circ\) and 45.0\(^\circ\). Fig. 5a and b represents the load–depth curves for \((\sigma_y = 800 \text{ MPa, } n = 5; \sigma_y = 420 \text{ MPa, } n = 2.6)\) and \((\sigma_y = 800 \text{ MPa, } n = 5; \sigma_y = 600 \text{ MPa, } n = 3.7)\), respectively. Elastic moduli of them are fixed as 200 GPa. Table 1 shows that gap of \(C\) values for three indenter angles. Although there is no significant difference in \(C\) between the two materials, \(\sigma_y = 800 \text{ MPa, } n = 5\) and \(\sigma_y = 420 \text{ MPa, } n = 2.6\), for the angle of the indenter \(\alpha = 70.3^\circ\), the difference becomes about 15% for \(\alpha = 45^\circ\). In contrast to the previous case, the two materials, \(\sigma_y = 800 \text{ MPa, } n = 5\) and \(\sigma_y = 600 \text{ MPa, } n = 3.7\), show 8% deviation of \(C\) for the indenter angle 70.3\(^\circ\) whereas the difference is negligible for \(\alpha = 45^\circ\). It means that two materials can be distinguished by using the two or more self-similar indenters. Note that when a lager indenter angle range is used, load–displacement curves for sets of arbitrary materials become quite separable. Based on this, we can obtain the indentation formula which converts load–depth curves to true stress–strain curves, and then suggest a dual (or plural) indentation algorithm based on FEA solutions for the material property evaluation. Table 2 shows representative material properties used in this paper, which cover the range of material property of general metals.

### 4. Indentation numerical approach based on finite element analysis solution

#### 4.1. Numerical formulas for young’s modulus evaluation

The slope of unloading curve, elastic recovery and contact area are related with Young’s modulus (Sneddon, 1965).
Pharr et al. (1992) reported that the unloading curve is nonlinear, and the initial unloading slope of curve has a close relation with Young’s modulus. Pharr et al. (1992) suggested the theoretical formula as follows:

\[
E = \frac{1 - \nu^2}{\beta d^2(1 - \nu^2)/E_i}
\]  

(8)

Here \(d\) is actual projected contact diameter considering the effect of pile-up/sink-in and \(S\) is initial unloading slope. \(\beta\) is the correction factor that depends on material properties and the indenter geometry. \(E_i\), \(\nu\), and \(E\) are Young’s modulus, Poisson’s ratio of the indenter and the specimen respectively. Lee et al. (2008) studied the variation of \(\beta\) with regression range and fitting function type. For regression of unloading slope, Lee et al. (2008) considered two regression functions and two unloading ranges: power law and linear fits, and 20% and 80% of unloading data. They showed that \(\beta\) may be regarded as almost constant for various indentation parameters such as Young’s modulus, yield strain and strain-hardening exponent and indenter tip-radius.

### Table 3

Comparison of computed material properties with a WC indenter to those given.

<table>
<thead>
<tr>
<th>(\sigma_0/E) (\sigma_s) (MPa)</th>
<th>(n)</th>
<th>Computed (E) (GPa)</th>
<th>Error (%)</th>
<th>Computed (\sigma_0) (MPa)</th>
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<td>1998.2</td>
<td>0.1</td>
<td>19.9</td>
<td>0.10</td>
<td></td>
</tr>
</tbody>
</table>
When $\beta$ is a constant, Lee et al. (2008) obtained four regression constants as follows:

\[
\begin{align*}
\beta &= 1.067; & \Delta P/P_{\text{max}} &= 20\%, & \text{power law regression} \\
1.101; & \Delta P/P_{\text{max}} &= 80\%, & \text{power law regression} \\
1.058; & \Delta P/P_{\text{max}} &= 20\%, & \text{linear regression} \\
0.987; & \Delta P/P_{\text{max}} &= 80\%, & \text{linear regression}
\end{align*}
\] (9)

Here $\Delta P (=P_{\text{max}} - P)$ is the difference between the maximum load at the maximum indentation depth and a chosen indentation load on the unloading curve (Fig. 1).

When $\beta$ is a function of Young’s modulus, $\beta$ is given as follows:

\[
\beta = c_1 + c_2 \left( \frac{E}{E_p} \right)
\]

\[
(c_1, c_2) = (1.051, 0.04223); \quad \Delta P/P_{\text{max}} = 20\%, \quad \text{power law regression}
\] (10)

\[
(c_1, c_2) = (1.084, 0.04576); \quad \Delta P/P_{\text{max}} = 80\%, \quad \text{power law regression}
\]

\[
(c_1, c_2) = (1.043, 0.04047); \quad \Delta P/P_{\text{max}} = 20\%, \quad \text{linear regression}
\]

\[
(c_1, c_2) = (0.976, 0.02196); \quad \Delta P/P_{\text{max}} = 80\%, \quad \text{linear regression}
\]

Lee et al. (2008) calculated the correction factor $\beta$ from Eq. (9) or (10), and obtain the Young’s modulus using Eq. (8). Suggested methods successfully provide the value of elastic modulus with maximum error of less than 5%.

### 4.2. Calculation of contact diameter

Lee et al. (2008) calculated the Young’s modulus by using Eq. (8) and projected contact diameter measured at unloading condition. In reality, it is difficult to measure the contact diameter; inaccurately obtained contact diameter can cause further error in the evaluation results. In this study, the $c^2$ values representing the amount of pile-up and sink-in were obtained via the FE simulation, and then the projected contact diameter was estimated. In this study, the $c^2$ is defined as follows:

\[
c^2 = \left( h_c/P_{\text{max}} + h_g \right) / \left( h_{\text{max}} + h_g \right) = f^e(\varepsilon_e, n)
\] (11)

Here $h_c$ is the actual contact indentation depth considering pile-up or sink-in (Fig. 2). Fig. 6 depicts the relation between $c^2$ and indentation depth for four yield strains. The almost same $c^2$ can be obtained for various indentation depths although a considerable $h_g$ (or tip radius $R$) is considered. The deviation of $c^2$ within the chosen $R/h_{\text{max}}$ ratios is less than 1%. When $h_g$ is not considered ($c^2 \equiv h_c/P_{\text{max}}/h_{\text{max}}$), the deviation of $h_c/P_{\text{max}}/h_{\text{max}}$ sharply increases with decreasing $R/h_{\text{max}}$ ratio. For $h = 15h_g$, it becomes more than 4%. Fig. 7 depicts the variations of $c^2$ with tip-radius $R$ ($R/h_{\text{max}} = 0, 0.5, 1.0, 2.0$) for various material properties. Fig. 7 shows that $c^2$ becomes uniform for the same indentation depth regardless of tip radius, $R/h_{\text{max}} = 0.5, 1.0, 2.0$. Hence, indentation depth and tip-radius does not effect on value of $c^2$.

Fig. 8 shows the distribution of the $c^2$ for various material properties from obtained FE analyses. We performed
FE analyses of a total of 56 cases (yield strain $\varepsilon_y$; 7 × strain-hardening exponent $n$: 8) with fixed Young's modulus to generate regression formulae (11). The WC conical indenter with a half-included angle of 70.3°/176° was used and the maximum indentation depth, $R/h_{\text{max}}$, was 0.5. In Fig. 8, $c^2$ value bigger than 1 means that pile-up occurs. The values of $c^2$ increase with strain hardening exponent and decrease with yield strain. We selected the $n$ and $e_0$ as the parameters of regression function of $c^2$ as described in Eq. (12). In this study, the coefficients of $c^2$ are first regressed on $n$ for each given $e_0$, and then obtained coefficients are regressed on $e_0$. Appendix A shows the regression functions and coefficients.

$$c^2 = f^e(e_0, n) = f^i(e_0)n^{-i} = (\gamma n^i e_0)n^{-i};$$
$$i, j = 0, 1, 2, 3, 4 \text{ (Appendix A)} \quad (12)$$

The regression lines of $c^2$ for various material properties are presented in Fig. 8.
Knowing $h_t (= h_{\text{max}} + h_g)$ and $c^2$, we can obtain projected contact diameter $d$ by the geometrical relation of conical indenter as follows:

$$d = 2c^2 h_t \tan \alpha = 2c^2 f'(e_o, n)(h_{\text{max}} + h_g) \tan \alpha$$  \hspace{1cm} (13)

Note that $d$ becomes a function of $c^2$, and thereby a function of material properties $e_o$ and $n$. Using Eqs. (12) and (13), we can express Eq. (8) as

$$E = \frac{(1 - v^2)}{2f(\tan \alpha)h_b f^c(e_o, n)/S_1 - (1 - v^2)/E}$$  \hspace{1cm} (14)

Here $S_1$ is initial slope of unloading curve obtained from 70.3° indenter.

4.3. Dual conical indentation techniques for material properties evaluation

We observed the variation of coefficient $C$ with material properties using two indenters of half-included angle 70.3°, 45° respectively. To minimize the tip radius effect, based on Lee et al. (2008)'s observation, the $C$ values were regressed by using the data ranged upper 50% of the maximum indentation depth $h_{\text{max}}$.

Fig. 9 represents the distribution of coefficient $C$ with yield strain and strain hardening exponent. In Fig. 9, the maximum value of $C/E$ is normalized to unity. As the difference of intersection angle of contours is getting smaller, it becomes harder to distinguish the material properties although two indenters with different angle are used. As the intersection angle contoured from the two indenters is getting bigger, the properties can be more easily indentified. We calculated the gradients of contours and the angles between the gradients as shown in Fig. 10. Since strain hardening exponents of materials are generally greater than 2, it would be fair to state that most materials can be distinguished by the dual indentation tests. Noted that Chen et al. (2007) did not show clearly the applicable range of multiple indentation method, but we definitely

---

**Fig. 16.** Comparison of stress–strain curves computed by two parameter regression on the entire strain range to those experimentally measured for (a) SCM4, (b) Al6061, (c) Brass, (d) SS400, (e) J2 and (f) API-X65.
demonstrated the sensitivity of the predicted material properties for the two indenter angles, $\alpha = 70.3^\circ$ and $45^\circ$.

Fig. 11a and b shows the distribution of coefficient $C$ with yield strain $\varepsilon_o$, strain hardening exponent $n$ when half-included angles of the tungsten carbide (WC) conical indenter are $70.3^\circ$ and $45^\circ$; the indentation depth $R/\text{h}_{\text{max}} = 0.5$. We performed FE analyses of a total of 294 cases (Young’s modulus $E$: 3 $\times$ yield strain $\varepsilon_o$: 7 $\times$ strain-hardening exponent $n$: 7 $\times$ half-included angle $\alpha$: 2). The values of coefficient $C$ obtained from FE analysis for various $\varepsilon_o$ and $n$ are regressed to dimensionless formula normalized with elastic modulus $E$. The regression method is the same as Eq. (12). The final regression function is a polynomial in the form of Eq. (15).

$$C_i/E = \sum_{j=0}^{k} (\delta_{ij}\varepsilon_o^j/n^j; \ i = 1, 2; \ j, k = 0, 1, 2, 3, 4) \quad \text{(Appendix A)}$$

Fig. 17. Errors of computed material property values (a) $E$, (b) $\sigma_o$ and (c) $n$ to those given (2% increases of $C_1$ and $C_2$).

Fig. 18. Errors of computed material property values (a) $E$, (b) $\sigma_o$ and (c) $n$ to those given (2% decreases of $C_1$ and $C_2$).
Here \(i = 1, 2\) represents 70.3° and 45° respectively. As \(C_1\) and \(C_2\) in Eq. (15) can be determined by the dual indentation tests, Eq. (15) becomes the nonlinear simultaneous equations for \(e_o\) and \(n\).

Substituting Eq. (14) into Eq. (15) leads to the followings:

\[
C_1 = Ef_C^C(e_o, n) - \frac{f_C^C(e_o, n)}{Mf_C^C(e_o, n)} - N;
\]

\[
F(e_o, n) \equiv \frac{f_C^C(e_o, n)}{Mf_C^C(e_o, n)} - C_1 = 0
\]

\[
C_2 = Ef_C^C(e_o, n) - \frac{f_C^C(e_o, n)}{Mf_C^C(e_o, n)} - N;
\]

\[
G(e_o, n) \equiv \frac{f_C^C(e_o, n)}{Mf_C^C(e_o, n)} - C_2 = 0
\]

\[
M = \frac{2(\tan \alpha)h_0}{S_1(1 - v^2)}, \quad N = \frac{(1 - v^2)}{E_1(1 - v^2)}.
\]

We calculate \(e_o\) and \(n\) which satisfy \(F = 0\) and \(G = 0\) of Eq. (16) using Newton–Raphson method. Based on the contact diameter at unloaded state, Lee et al. (2008) calculated \(\beta = 1.058\) by linear fitting for the 20% unloading range. Meanwhile, Dao et al. (2001) used \(\beta = 1.06\) in conical indentation. However, it is a very difficult that we measure the accurate contact diameter at unloading state. We can predict the actual contact diameter at unloading state, and calculate the more accurate \(\beta\). In Eq. (8), we used \(\beta\) obtained by linear fit for 20% of unloading data. Fig. 12 shows the distribution of \(\beta\) obtained with linear fitting for various values of \(e_o\) and \(n\). The deviation of \(\beta\) is less than 2% irrespective of chosen material properties, \(\beta\) can be regarded as a constant. We adopt the mean value of \(\beta = 1.068\) which is obtained in \(E = 200\) GPa. Our \(\beta\) value is quite consistent with the other suggested material properties. Furthermore, it is very accurate for widely \(e_o\) and \(n\) scope.

Using Eqs. (16), we established an evaluation program providing material properties and corresponding stress–strain curve from load–displacement curves of dual conical indentation tests as shown in Fig. 13. First, we obtain the Kick’s law coefficient \(C_1\) and \(C_2\) from load–displacement curves using two different indenters. We first set up the initial value of \(e_o\) and \(n\); then, we find the new \(e_o\) and \(n\) using Eq. (16). Substituting this value for Eq. (14), we calculate the Young’s modulus \(E\). Although evaluation program providing material properties is based on \(E = 200\) GPa, the program is still effective for other elastic moduli, because the relative ratio (=yield strain, \(e_o\)) is more important indentation parameter than absolute values of elastic modulus and yield strength (Fig. 14). The obtained material properties are compared with those used for finite element analyses in Table 3. The average error of predicted material properties is less than 1%, and maximum error is about 5%. Fig. 15 shows predicted and given material curves for various material properties. The solid line is the material curve used for FEA, and the symbol is the predicted stress–strain curve. These figures show the validation of new approach for various values of \(n\) and \(e_o\).

In this study, we calculated the elastic modulus \(E\) by using Eq. (8). Here, the values for \(S\) and \(d\) were obtained from FE analysis using the 70.3° indenter. The projected contact diameter is a function of material properties, tip-radius and indenter angle. The contact diameter \(d\) as well as \(S\) depends on the indent angle \(\alpha\). If the contact diameters for various material properties using the 45° indenter are obtained from FE analyses, we can also predict the \(E_{45^\circ}\). Although the predicted values for \(E_{45^\circ}\) are equal, their values vary slightly, although the difference is virtually negligible, due to errors

<table>
<thead>
<tr>
<th>Table 7</th>
</tr>
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<tr>
<td>Sensitivity of the estimated mechanical properties to variation in the Kick’s law coefficient.</td>
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</table>

<table>
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<tr>
<th>Dual conical indentation</th>
<th>0% in (C_1)</th>
<th>+2% in (C_{1.2})</th>
<th>−2% in (C_{1.2})</th>
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<td>(E)</td>
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<td>1.13</td>
<td>0.78</td>
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<tr>
<td>(\sigma_o)</td>
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<td>4.80</td>
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<tr>
<td>(n)</td>
<td>1.42</td>
<td>3.68</td>
<td>7.11</td>
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<table>
<thead>
<tr>
<th>Table 8</th>
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<tr>
<td>Comparison of (P_{\text{max}}) values for various values of friction coefficient.</td>
</tr>
</tbody>
</table>

\[
E = 200\ \text{GPa}, \ \sigma_o = 400\ \text{MPa}, \ n = 10, \ \frac{R}{h_{\text{max}}} = 0.5
\]

\[\alpha (°) \quad f \quad P_{\text{max}} \quad \text{Gap} (%)\]

\[
\begin{array}{cccc}
70.3 & 0.1 & 129.9 & 1.2 \\
0.2 & 128.5 & 0.1 \\
0.3 & 128.4 & - \\
0.5 & 128.4 & 0.1 \\
45 & 0.1 & 25.7 & 7.6 \\
0.2 & 27.2 & 2.2 \\
0.3 & 27.8 & - \\
0.5 & 27.7 & 0.4
\end{array}
\]

**Fig. 19.** \(P/Eh_{\text{max}}^2\) vs. \(h/h_{\text{max}}\) curves for four different friction coefficients.
in the numerical calculation. However, the important thing is their accuracy. The average error of the predicted $E_{45}/C_{176}$ is also less than 1% (Table 4). When we evaluate material properties using diamond indenter ($E_I = 1000$ GPa, $m_I = 0.07$), the obtained material properties are almost similar to those of tungsten carbide indenter ($E_I = 537$ GPa, $m_I = 0.24$) (Table 5). The blunted indenter tip may cause nontrivial problems in evaluating material properties. However, when tip-radii of indenters varies ($R/h_{\text{max}} = 0$, 0.5, 1, 1.5), the average errors for $e_0$ and $n$ are less than 2% and 5% (Table 6). When $C$ is calculated, the gap $h_g$ between indenters with zero and finite tip-radii can be determined by Eq. (2). Therefore, the finite tip-radius effect can be eliminated in the loading curve.

4.4. Evaluation of material properties by using experimentally obtained stress–strain curves

We performed the tensile and compression tests according to ASTM E8 to obtain tensile material properties. Using an MTS tensile testing system, we carried out tensile tests for round bar specimens (diameter: 6 mm) by 3 mm/min. We tested six different metallic materials: SCM4, Al6061, Brass, J2, SS400, and API-X65. We carried out the indentation test via FE analysis with the tensile testing data inputted. The stress–strain curves obtained from ten-

Table 9
Comparison of computed material property values to those given for various values of friction coefficient.

<table>
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<tr>
<th>$\sigma_0/E$</th>
<th>$n$</th>
<th>$f$</th>
<th>Computed $\sigma_0/E$ (MPa/GPa)</th>
<th>Error (%)</th>
<th>Computed $n$</th>
<th>Gap</th>
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<td>32.8/2.8</td>
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<td>0.8</td>
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<tr>
<td></td>
<td>0.2</td>
<td></td>
<td>432.0/196.4</td>
<td>8.0/1.8</td>
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</tr>
<tr>
<td></td>
<td>0.3</td>
<td></td>
<td>404.8/200.4</td>
<td>1.2/0.2</td>
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<td>0.0</td>
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<tr>
<td>0.004</td>
<td>3</td>
<td>0.1</td>
<td>993.9/193.7</td>
<td>24.2/3.2</td>
<td>3.8</td>
<td>0.8</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
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<td>844.1/196.4</td>
<td>5.5/1.8</td>
<td>3.2</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>0.3</td>
<td></td>
<td>797.8/199.9</td>
<td>0.3/0.0</td>
<td>3.0</td>
<td>0.0</td>
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<td>0.006</td>
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<td>19.2/3.3</td>
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<td>3.1</td>
<td>0.1</td>
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<tr>
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<td>0.3</td>
<td></td>
<td>1595.0/199.9</td>
<td>0.3/0.2</td>
<td>3.0</td>
<td>0.0</td>
</tr>
<tr>
<td>0.010</td>
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<td>0.1</td>
<td>1972.1/195.3</td>
<td>12.0/2.4</td>
<td>7.1</td>
<td>2.1</td>
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Fig. 20. Overall mesh design for 1/6 Berkovich indenter.
sile and indentation are compared in Fig. 16. Solid lines are obtained by tensile tests, the symbols are obtained by dual indentation tests. The predicted stress–strain curves almost agree well with given stress–strain curve except for Brass. The difference of yield strength between predicted (108 MPa) and given (156 MPa) for Brass are large. Due to the limitation of power law expression of stress–strain curve, the difference of yield strength between predicted and given is large in some materials not obeying power law. Moreover, a material having a large Lüders strain such as API-X65 shows a discrepancy in the early plastic region of stress–strain curve (Hyun et al., 2008).

5. Sensitivity analysis

Load–displacement curves can vary depending on the various conditions of experiment. It is therefore necessary to examine the sensitivity of evaluated material properties. Chollacoop et al. (2003), Swaddiwudhipong et al. (2005) and Harsono et al. (2009) reported that experimental scatter for Kick’s law coefficient $C$ is about 2%. We thus changed the value of $C \pm 2\%$ obtained from FEA with half-included angles $70.3^\circ$ and $45^\circ$, and estimated the values of material properties. We confirmed the errors of predicted material properties with Kick’s law coefficients for various material properties, $\varepsilon_o = 0.001, 0.002, 0.004, 0.006, 0.008$ and $n = 2, 3, 5, 7, 10, 13, 20$. Fig. 17a–c depicts the error of elastic modulus, yield strength, and the gap of strain-hardening exponent, respectively when the $C_1$ and $C_2$ are artificially increased simultaneously. On the other hand, Fig. 18a–c show the error of predicted material properties when the $C_1$ and $C_2$ are decreased simultaneously. In Figs. 17a and 18a, the maximum errors of predicted $E$ are less than 3% regardless of variation in $C$. However, $\sigma_y$ shows the rather strong sensitivity to variation in $C$ than $E$ in Figs. 17b and 18b. When we increase or decrease the values of $C_1$ and $C_2$ simultaneously, mean errors of estimated values are less than 10%. However, when $C_1$ is increased (or decreased) and $C_2$ is decreased (increased), the average of the error of the yield stress becomes about 20%. It should be mentioned that an indentation system will tend to only overestimate or underestimate for the two consecutive tests because the test condition will be similar. Therefore, we can conclude that the proposed method has weak sensitivity to perturbation in input parameters, $C_1$, $C_2$. Table 7 shows the average errors and their standard deviations (SD).

Fig. 19 shows the load–depth curves for various values of friction coefficient ($f = 0.1, 0.2, 0.3$ and 0.5). Table 8 shows the gap of maximum load $P_{\text{max}}$ with $P_{\text{max}|f=0.3}$ as the base value. The load–depth curves for $f = 0.2–0.5$ are similar to that for $f = 0.3$. However, the load–depth curve for $f = 0.1$ is notably different, which may result in significant errors in material properties. We thus verify that the evaluation program established with $f = 0.3$ is valid for $f = 0.1, 0.2$. Table 9 represents predicted material properties for $f = 0.1, 0.2$ and 0.3. The errors of predicted elastic modulus are less than 4% regardless of friction coefficient.
For \( f = 0.2 \), the errors in \( \sigma_o \) are less than 10%. For \( f = 0.1 \), however, the maximum error in \( \sigma_o \) is about 30%, and the errors in \( n \) also become significant. The proposed method based on \( f = 0.3 \) is valid for \( f > 0.2 \), and therefore further study is required.

### 6. Difference between conical and Berkovich indenters

The 70.3° conical indenter theoretically has the same cross-sectional area-to-depth ratio as Berkovich indenter. Due to difference of actual contact area and contact stiffness, the load–depth curves of both Berkovich and its equivalent conical indenters are not identical (Shim et al., 2007). We compared the load–depth curves obtained from 2D conical indenter and 3D Berkovich indenter. Considering geometrical symmetry, we made the 1/6 Berkovich indenter model using the 8-node brick element C3D8 (ABAQUS, 2007). FE model consists of about 36,000 8-node brick elements (C3D8) and 45,000 nodes (Fig. 20). In an inclination surface of 60° for x axis, the ratio of displacements between x and y axis should be maintained as 1 : \( \sqrt{3} \). The indenter downs to penetrate the material with the bottom of the specimen fixed.

When \( E \) and \( R/h_{\text{max}} \) are 70 GPa and 0, respectively, we observed the variations of the load–depth curves with strain hardening exponents \( (n = 3, 13) \) and yield strains \( (\varepsilon_o = 0.001, 0.002, 0.003, 0.004) \) (Fig. 21). As the \( n \) decreases and the \( \varepsilon_o \) increases, the difference between the load–depth curves obtained from conical and Berkovich indenters increases. The maximum differences of \( C/E \) and \( P_{\text{max}} \) values between conical and Berkovich are about 5% (Fig. 22). Therefore, it should be investigated the causes of the difference, and further study on the theory of Berkovich indentation is needed.

### 7. Summary

In this paper, we suggested the reverse algorithm for evaluation of material properties using the dual conical indentation. The proposed method successfully provides the value of material properties with the maximum error of less than 5%, regardless of tip-radius and materials properties. As mentioned in review section, while other researchers have used the concept of the representative, we provide the indention formulae through the multi regression of material properties, \( \sigma_o/E = (\varepsilon_o) \) and \( n \) without the defination of representative values.

We observed the effect of conical indenter angle on indentation load–depth curves via finite element analyses. We investigated then the relations among elastic modulus, yield strength, strain hardening exponent, and load–depth curve, and we confirmed that yield strain and strain...
hardening exponent are major parameters. We presented then the Kick’s law coefficient with indenter angle as function of yield strain and strain hardening exponent. To calculate the contact diameter, we introduced the dimensionless parameter \(c_0\), which is defined as the ratio of the tip-corrected indentation contact depth to the tip-corrected maximum depth, and then expressed it as a function of material properties. Using the \(c_0\) and geometrical relation of conical indenter, we obtained numerical formula for evaluation of Young’s modulus. Finally, we suggested an effective numerical approach for evaluation of yield strength and strain-hardening exponent using the dual conical indentation through the combination of numerical formulae. The proposed method is valid in wide range of materials properties, especially which have frictional coefficient bigger than 0.2, and the method shows weak sensitivity to perturbation of input parameters, \(C_1\), \(C_2\). In addition, the difference of load–depth curves obtained from a conical indenter and Berkovich indenter is discussed. Further studies on the effect of friction coefficient and the types of indenters are required.

Acknowledgments
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Appendix A

The coefficient values of regression functions of Eqs. (11) and (14) (see Tables A.1 and A.2, Figs. A.1 and A.2).

References


Fig. A.2. The regression curves of \(f_{c}^{2}\) in Eq. (14) vs. yield strain.


