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Further analysis of indentation loading curves: Effects of tip rounding on mechanical property measurements

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The effects of indenter tip rounding on the shape of indentation loading curves have been analyzed using dimensional and finite element analysis for conical indentation in elastic-perfectly plastic solids. A method for obtaining mechanical properties from indentation loading curves is then proposed. The validity of this method is examined using finite element analysis. Finally, the method is used to determine the yield strength of several materials for which the indentation loading curves are available in the literature.

I. INTRODUCTION

Indentation experiments have been performed for nearly one hundred years for measuring the hardness of materials.[1] Recent years have seen increased interest in indentation because of the significant improvement in indentation equipment and the need for measuring the mechanical property of materials on small scales. With the improvement in indentation instruments, it is now possible to monitor, with high precision and accuracy, both the load and displacement of an indenter during indentation experiments in the respective micro-Newtons and nanometer ranges.[2–4] In addition to hardness, basic mechanical properties of materials, such as the Young's modulus, yield strength, and work hardening exponent, may be deduced from the indentation load versus displacement curves for loading and unloading. For example, the hardness and Young's modulus may be calculated from the peak load and the initial slope of the unloading curves using the method of Oliver and Pharr[5] or that of Doerner and Nix.[6] Finite element methods have also been used successfully to extract the mechanical properties of materials by matching the simulated loading and unloading curves with that of the experimentally determined ones.[7–10]

Recently, many attempts have been made to better understand indentation loading curves. For example, several empirical formulae have been proposed for the loading curves in terms of Young's modulus and hardness.[11,12] Loading curves have also been discussed using energetic considerations of reversible and irreversible parts of the indentation-induced deformations.[13] Using dimensional analysis and finite element calculations, we have recently derived scaling relationships for indentation into elastic-perfectly plastic solids using conical indenters.[14,15]

In this paper, we apply the scaling relationships of indentation to determine the mechanical properties of materials. We first review, in Sec. II, the scaling relationships for ideally sharp conical indenters, followed by an analysis of the effects of indenter tip rounding on the shape of indentation loading curves in Sec. III. We will then propose, in Sec. IV, a method for extracting the mechanical properties from indentation loading curves obtained using conical indenters with rounded tips. The validity of this method is examined using finite element analysis. Finally in Sec. V, the method is used to determine the yield strength of several materials for which the indentation loading curves by pyramidal indenters are available in the literature. This new indentation method allows the measurement of certain mechanical properties of solids without measuring or estimating the contact area.

II. DIMENSIONAL AND FINITE ELEMENT ANALYSIS OF INDENTATION LOADING CURVES OBTAINED USING IDEALLY SHARP CONICAL INDENTERS INDENTING ELASTIC-PERFECTLY PLASTIC SOLIDS

We have shown[14,15] that for an ideally sharp, three-dimensional, rigid, frictionless, conical indenter of a given half angle (θ) (Fig. 1) indenting normally into an elastic-perfectly plastic solid characterized by Young's modulus (E), Poisson's ratio (ν), and yield strength (Y),

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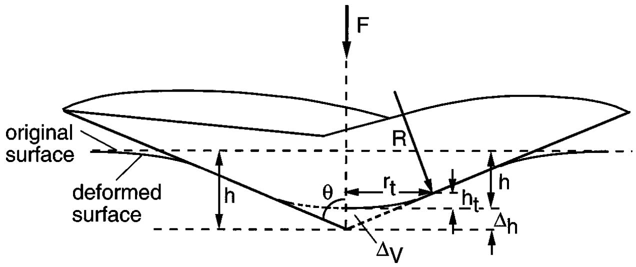

FIG. 1. Illustration of ideally sharp conical indenter and that with a spherical tip.

the load (F) on the indenter is a function of E, ν, Y, and the indenter displacement (h) and is given by:

$$F = Eh^2 \Pi\left(\frac{Y}{E}, \nu, \theta\right), \qquad (1)$$

where $\Pi = F/Eh^2$ is a dimensionless function of three-dimensionless parameters Y/E, ν, and θ. Equation (1) shows that the load on the indenter (F) is proportional to the square of the indenter displacement (h). Furthermore, the problem of determining the loading curves for conical indentation in elastic-perfectly plastic solids is reduced to determining a function of three parameters Y/E, ν, θ, instead of a function of *five* parameters, $F = F(E, Y, \nu, \theta, h)$.

Since in practice indenters are usually made for few specific angles, it is sufficient to determine the dimensionless function, $\Pi(Y/E, \nu, \theta)$, for the indenter angles of interest. Following the work of Bhattacharya and Nix[7] and Laursen and Simo,[8] we consider $\theta = 68°$, for it gives approximately the same volume to depth relation as that of ideally sharp Vickers indenters[7,8] and, to a good approximation, that of ideally sharp Berkovich indenters ($\theta = 70.3 \ldots$ degrees).[10] Finite element calculations using ABAQUS[16] have been carried out to evaluate the function $\Pi(Y/E, \nu, \theta)$ for $\theta = 68°$. To simplify notation, $\Pi(Y/E, \nu)$ is used instead of $\Pi(Y/E, \nu, 68°)$. The dependence on indenter angle is implied. The finite element model has been described in detail previously.[14,15]

Figure 2 depicts $F/Eh^2 = \Pi(Y/E, \nu)$ versus Y/E for two values of ν. For a given ν $\Pi(Y/E, \nu)$, is a nonlinear, single-valued function of Y/E. Furthermore, it was noticed[14] that the dimensionless function $\Pi(Y/E, \nu)$ could be expressed, to a good approximation, as a product of two-dimensionless functions $f(\nu)$ and $\Pi^*(Y/E)$,

$$\frac{F}{Eh^2} = \Pi(Y/E, \nu) \approx f(\nu)\Pi^*(Y/E). \qquad (2)$$

Figure 3 depicts $F(1 - \nu^2)/Eh^2$ versus Y/E. The two quantities, $F(1 - \nu^2)/Eh^2$ and Y/E, lie approximately on a single curve, suggesting that

$$\frac{F}{Eh^2}(1 - \nu^2) \approx \Pi^*(Y/E). \qquad (3)$$

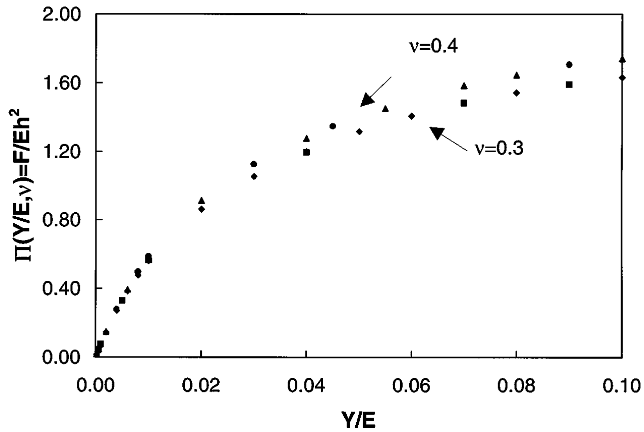

FIG. 2. Scaling relationships between F/Eh^2 and Y/E.

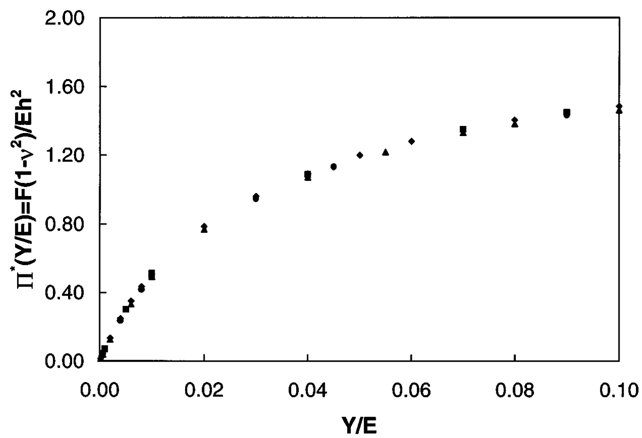

FIG. 3. An approximate scaling relationship between $F(1 - \nu^2)/Eh^2$ and Y/E.

Equations (1) and (3) offer the possibility for extracting materials properties from indentation loading curves. Because the load, F, and displacement, h, can be determined experimentally, Eq. (1) shows that Y/E can be determined from loading curves if ν is known. Consequently, Y (or E) can be determined provided that E (or Y) and ν are known. Since $\Pi(Y/E, \nu)$ does not change significantly for typical values of ν ($0.25 < \nu < 0.35$), an estimated Y (or E) may be obtained from loading curves by using a typical value of ν (i.e., 0.3) if E (or Y) is known. If ν is known, Y (or E) can also be evaluated using the approximate Eq. (3) and the known values for E (or Y). In practice, E is usually more easily obtained than Y by a variety of measurement techniques. Equation (3) shows that Y can then be obtained from indentation loading curves without the need of measuring hardness. This is useful because hardness requires the measurement or estimation of the contact area under load, which may be difficult to obtain. Furthermore, the conversion of hardness to yield strength is not always straightforward. However, it must be emphasized that, in principle, loading curves obtained using ideally sharp conical (or pyramidal) indenters alone cannot uniquely determine both E and Y. In order to use the above procedure in practice, it is necessary to consider the effect of indenter tip imperfection encountered in indentation experiments.

III. INFLUENCE OF TIP GEOMETRIC IMPERFECTION ON THE SHAPE OF INDENTATION LOADING CURVES

In practice, the indenter tips are not ideally sharp. This imperfection may be modeled by a conical indenter with a spherical tip. The side of the cone is assumed to be tangent to the sphere as shown schematically in Fig. 1. The tip geometry is determined by the tip radius, R, and the indenter half angle, θ. Specifically, the tangent point is given by:

$$\frac{h_t}{R} = 1 - \sin\theta \overset{\theta=68^\circ}{=} 0.0728\,, \tag{4}$$

$$\frac{r_t}{R} = \cos\theta \overset{\theta=68^\circ}{=} 0.375\,. \tag{5}$$

The distance between the tips of the two indenters, Δh in Fig. 1, is given by

$$\frac{\Delta h}{R} = \frac{1}{\sin\theta} - 1 \overset{\theta=68^\circ}{=} 0.0785\,. \tag{6}$$

Neglecting sinking-in and piling-up of surface profiles around the indenter, loading curves should follow that of spherical indentation when $h/R < 0.073$ and approach that of ideally sharp conical indentation when $h/R \gg 0.073$. When h and R are of the same order of magnitude, which is frequently encountered in practice, loading curves are expected to deviate from that for spherical or ideally sharp conical indenters.

For indentation in elastic-perfectly plastic solids using rigid conical indenters (e.g., $\theta = 68°$) with a spherical tip, the force, F , on the indenter is a function of E, Y, ν, h, as well as R. Dimensional analysis yields

$$F = Eh^2 \Pi_R\left(\frac{Y}{E}, \nu, \frac{h}{R}\right), \tag{7}$$

where Π_R is a dimensionless function of three dimensionless parameters Y/E, ν, and h/R (the dependency on indenter angle is implied). Consequently, the load, F, is, in general, not proportional to the square of the displacement, h. It may depend on h/R through the dimensionless function Π_R.

Finite element calculations were carried out to illustrate the changes in loading curves with varying R. As seen from Fig. 4, the deviation from that of ideally sharp conical indenter increases with decreasing h/R. Consistent with common experiences the force required to move the indenter to the same depth h increases with

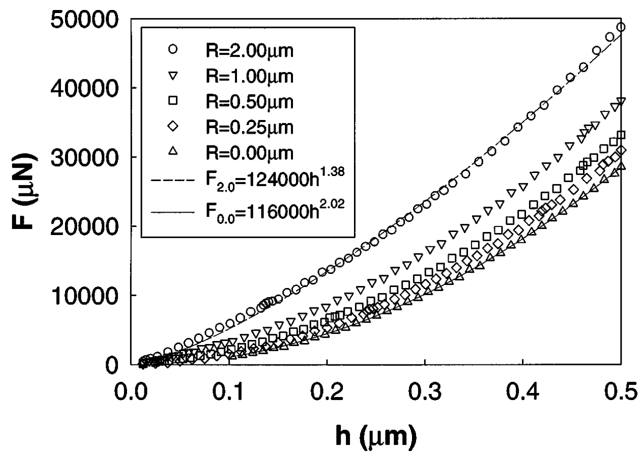

FIG. 4. Effects of indenter tip radius on the shape of indentation loading curves. The indentation loading curves are calculated using a finite element method for elastic-perfectly plastic solids characterized by $E = 200$ GPa, $\nu = 0.3$, and $Y = 2$ GPa.

increasing R. Furthermore, the force on the indenter can indeed deviate significantly from the square of the indenter displacement h, when h and R are of the same order of magnitude.

The effect of tip radius can be more generally illustrated in the dimensionless form by plotting F/Eh^2 versus h/R as shown in Fig. 5. According to Eq. (7), the two quantities, F/Eh^2 and h/R, should lie on a single curve for a given Y/E and ν. In particular, they should lie on a single curve for the same homogeneous material (E, Y, and ν fixed) indented by conical indenters of different tip radii, R. These features are demonstrated in Fig. 5. It is evident from both Figs. 4 and 5 that a significant deviation from ideally sharp conical indenters can be expected when $h/R < 1$. Furthermore, Π_R $(Y/E, \nu, h/R)$ approaches Π $(Y/E, \nu)$, when $h/R \gg 2$. Quantitatively, the deviation from that of ideally

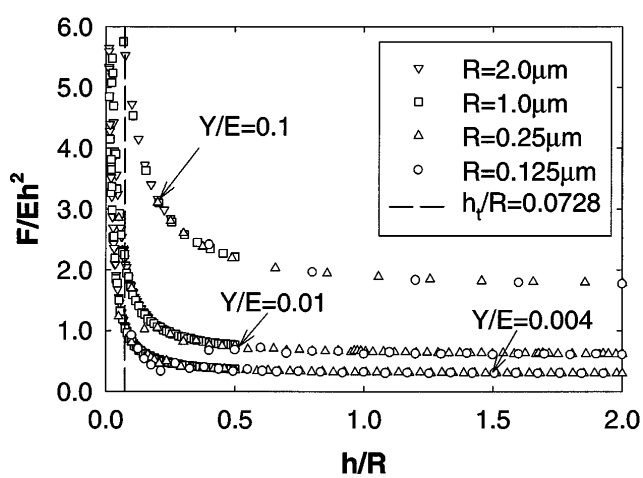

FIG. 5. Effects of indenter tip radius shown as dimensionless parameter F/Eh^2 versus h/R for several typical values of Y/E and $\nu = 0.3$.

sharp conical indenters at a given h/R also depends on mechanical properties through the ratio Y/E. This is due to the fact that the degree of sinking-in and piling-up, which affects the location of the contacts, is determined by Y/E.[14,15]

IV. A NEW METHOD FOR ANALYZING INDENTATION LOADING CURVES

First, we note that the difference of the indented volume, ΔV, shown in Fig. 1, between that of the ideally sharp conical indenter (e.g., $\theta = 68°$) and that with a spherical tip, is about $6.0 \times 10^{-3}\ R^3$. The volume of the total indented volume of the ideally sharp conical indenter, V, is on the order of $6h^3$. The ratio, $\Delta V/V$, is about $10^{-3}\ (R/h)^3$. This ratio is less than 1%, even when $h/R = 0.5$. Assuming the contribution to force, ΔF, due to ΔV is small, indentation by a conical indenter with spherical tip may be viewed as indentation by an ideally sharp indenter with its initial tip position shifted by Δh shown in Fig. 1. Under this assumption we expect the loading curves to follow

$$F + \Delta F = E(h + \Delta h)^2 \Pi\left(\frac{Y}{E}, \nu\right), \qquad (8)$$

in which the dimensionless function $\Pi\ (Y/E, \nu)$ is the same as that for the ideally sharp conical indenters. Equation (8) suggests that the loading curves for conical indenters with a spherical tip can be approximated by a second-order polynomial,

$$F = c_0 h^2 + c_1 h + c_2\,, \qquad (9)$$

where the coefficient c_0 depends only on the property of materials (E, ν, Y), c_1 and c_2 are functions of tip radius and materials properties. Specifically,

$$c_0 = E\Pi(Y/E, \nu)\,, \qquad (10)$$

and, using Eq. (6),

$$c_1 = 2c_0 \Delta h = 2E\Pi(Y/E, \nu)\left(\frac{1}{\sin\theta} - 1\right)R\,. \qquad (11)$$

Fig. 6 shows the fits using Eq. (9) to the calculated indentation loading curves shown in Fig 4. Excellent agreements are evident for a wide range of h/R, confirming that the loading curves can be approximated by the second-order polynomials given by Eq. (9).

Table I summarizes the fitting parameters c_0/E and the tip radius, R, evaluated using Eq. (11) for three values of Y/E. The fitting parameter c_0/E agrees, to less than 3%, with that for the ideally sharp conical indenter for $h/R > 0.5$ and less than 17% for $0.25 < h/R < 0.5$ (Table I), suggesting that the parameter c_0/E is indeed relatively insensitive to the radius within the range of h/R considered. The tip radius, R, evaluated

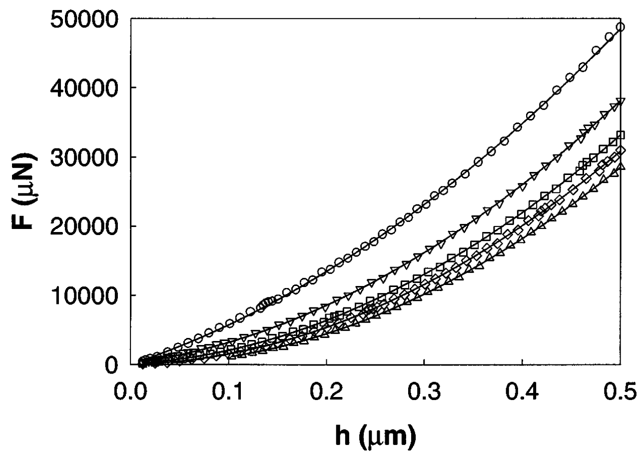

FIG. 6. A numerical fit to the indentation loading curves shown in Fig. 4 using second-order polynomials.

TABLE I. Examples of using second-order polynomials to model indentation loading curves for 68° half angle conical indenters with finite tip radii, R. The input parameters to the finite element calculations are $E = 200$ GPa and $\nu = 0.3$ together with several values of Y and tip radii R_{mod}. The three Y/E values represent the typical range of Y/E encountered in materials. The final indentation depth is 0.5 μm. The range of h/R is, therefore, between 0.25 and 2.0. The coefficients of the second order polynomial are used to calculate tip radius, R, and the dimensionless function $\Pi\ (Y/E, \nu) = c_0/E$. It is evident that the value of c_0/E is relatively insensitive to the indenter tip radius.

	Y/E					
	0.10		0.01		0.004	
R_{mod} (μm)	R (μm)	c_0/E	R (μm)	c_0/E	R (μm)	c_0/E
0.00	0.0	1.66	0.0	0.59	0.0	0.28
0.25	0.18	1.68	0.21	0.58	0.20	0.28
0.50	0.44	1.68	0.46	0.58	0.51	0.28
1.00	1.1	1.62	1.1	0.57	1.1	0.27
2.00	3.3	1.42	3.0	0.50	3.1	0.24

using Eq. (11), also approximately agrees with that given to the finite element models (Table I).

Based on the above analysis, we propose a new method for determining yield strength, Y, and indenter tip radius, R, using indentation loading curves together with known E and ν:

(1) Experimental loading curve is fitted with a second-order polynomial, Eq. (9), and the coefficients, c_0 and c_1, are determined.

(2) Divide c_0 by E and obtain $\Pi\ (Y/E, \nu) \approx c_0/E$.

(3) Determine Y/E, and consequently Y, using the scaling relationship $\Pi\ (Y/E, \nu)$ given by Fig. 2 or from the approximate scaling relationship $(1 - \nu^2)\Pi^*$ (Y/E) given by Fig. 3 together with either a known or representative value for ν.

(4) Determine the tip radius using c_0, c_1, and Eq. (11), i.e., $R = c_1/[2c_0(1/\sin\theta - 1)]$.

A similar method can obviously be used to determine E if Y and ν are known.

We would like to point out that based on empirical observations[13,17,18] and analysis of purely elastic contacts,[19,20] several authors have previously suggested the use of second-order polynomials [Eq. (9)] to describe indentation loading curves. However, the physical meaning for the coefficients of the second-order polynomial has not, to the best of our knowledge, been given explicitly as in Eqs. (10) and (11). These equations form the basis for this newly proposed analysis method for conical indentation into elastic-perfectly plastic solids.

V. ANALYSIS OF EXPERIMENTAL LOADING CURVES OBTAINED USING PYRAMIDAL INDENTERS

Although the above analysis applies strictly to indentation into elastic-perfectly plastic solids using rigid conical indenters, we demonstrate here that the method could be used to extract mechanical properties of real materials from indentation measurements using pyramidal indenters such as Vickers and Berkovich types.

Like the ideally sharp conical indenters, the ideally sharp pyramidal indenters are also geometrically self-similar and their shape is determined by a set of angles denoted by Θ. For a rigid pyramidal indenter of given shape indenting into elastic-perfectly plastic solids, the force on the indenter is, therefore, determined by E, ν, Y, and Θ. Dimensional analysis can then be carried out the same way as that for conical indentation:

$$F = Eh^2 \Pi\left(\frac{Y}{E}, \nu, \Theta\right), \tag{12}$$

where $\Pi\ (Y/E, \nu, \Theta)$ is a dimensionless function. The conclusions reached for conical indenters remain true: The force is proportional to the square of the indenter displacement; and the proportionality factor is only a function of Y/E and ν for a given indenter shape (i.e., fixed Θ). Three-dimensional finite element calculations are obviously needed to determine the function $\Pi\ (Y/E, \nu, \Theta)$ for the relevant shapes of pyramidal indenters. Such calculations, though computationally intensive, are within the current capabilities of commercial finite element software and have indeed been performed.[9]

However, since Eqs. (1) and (12) are the same in form, the results for conical indentation should capture the essential features of pyramidal indentation within the context of elastic-plastic deformation. Furthermore, previous finite element calculations for conical indention have shown good agreements with experimental indentation results obtained using pyramidal indenters, provided that the indented volume to indenter displacement relationship is the same in both cases.[7–10] In particular, the results of Vickers and Berkovich indentation are similar to that of conical indentation with a 68° and 70.3° half angle, respectively. Consequently, the numerical values

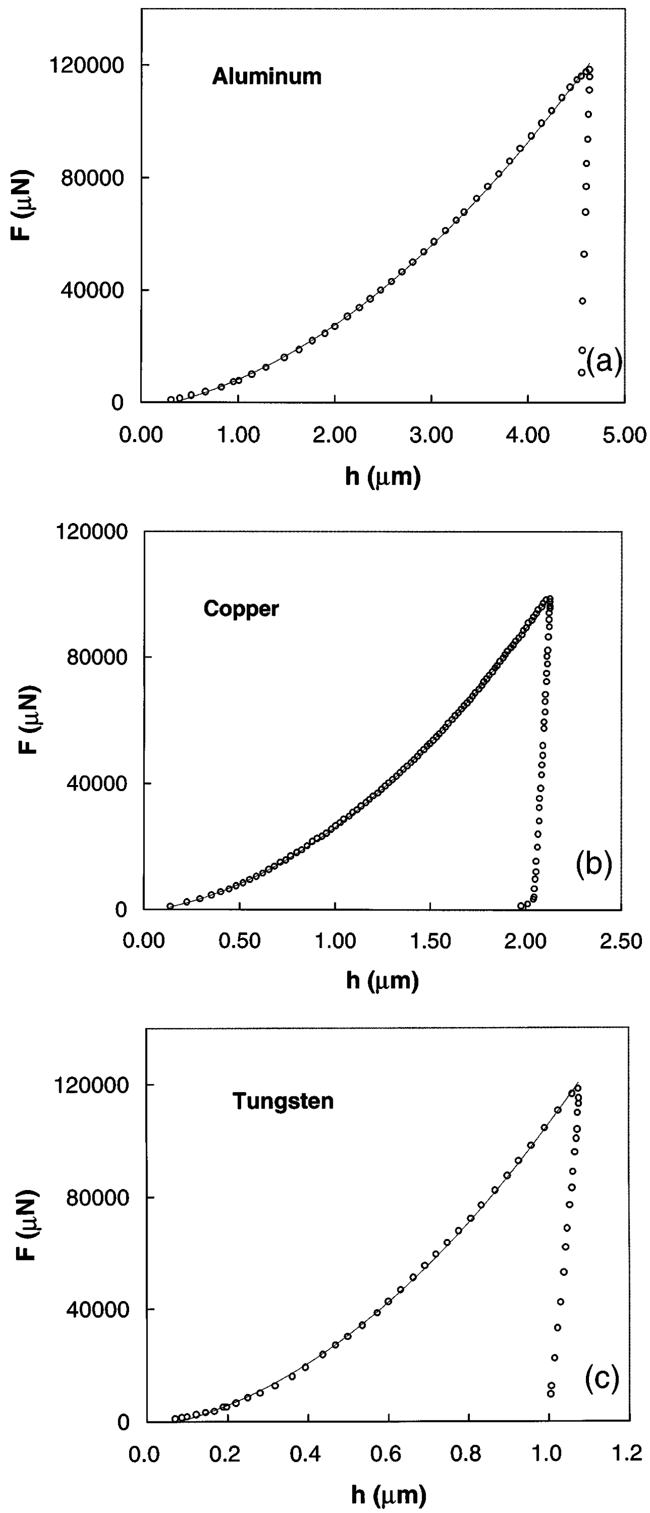

FIG. 7. A numerical fit using second-order polynomials to the experimental loading curves reported in the literature for (a) aluminum, (b) copper, and (c) tungsten.

of $\prod (Y/E, \nu)$ or $\prod^* (Y/E)$ for 68° conical indenters may be used to analyze Vickers and, as an approximation, Berkovich indentation loading curves. As a further approximation, the finite elastic constants of the diamond indenter is taken into account by substituting $E/(1 - \nu^2)$ in Eq. (3) by the reduced modulus commonly defined as[21]: $1/E^* = (1 - \nu^2)/E + (1 - \nu_i^2)/E_i$, where $E_i =$ 1140 GPa and $\nu_i = 0.07$ are Young's modulus and Poisson's ratio of the diamond indenters, respectively.

Several experimental loading curves for Al, W, and Cu obtained from the literature[5,12] have been digitized and replotted in Figs. 7(a)–7(c). These curves were obtained using pyramidal-shaped diamond indenters (Berkovich or Vickers). Figure 7 shows that the agreement between the experimental data and the fit using Eq. (9) is very good. Using Eqs. (1), (3), and (10), and the established values for E and ν from literature, the yield strength of the materials is calculated. Table II summarized the results together with literature values for Y. The agreement is reasonable considering the simplicity of the elastic-perfectly plastic model and the lack of information about the history of the materials used in actual indentation experiments.

VI. SUMMARY

The effects of indenter tip rounding on the shape of indentation loading curves have been analyzed using dimensional and finite element analysis for conical indentation in elastic-perfectly plastic solids. A method for obtaining mechanical properties from indentation loading curves has been proposed. The method consists of the following:

(i) Measuring loading curves using conical or pyramidal indenters.

(ii) Fitting indentation loading curves with second-order polynomials.

(iii) Obtaining yield strength using the coefficients of the second-order polynomials, scaling relationships for conical indentation, and known values of Young's modulus and Poisson's ratio.

TABLE II. An example of using the proposed method to estimate the yield strength of solids from indentation loading curves.

Materials	E (GPa)	ν	Y_{lit} (MPa)	Y (MPa)
Al	70[1]	0.33[1]	110[2]	70
Cu	115[1]	0.345[1]	320[2]	350
W	360[1]	0.2[1]	1800[2]	1400

[1] J. M. Gere and S. P. Timoshenko, *Mechanics of Materials,* 2nd ed. (PWS-KENT, Boston, 1984), p. 742.
[2] E. Rabinowicz, *Friction and Wear of Materials* (Wiley, New York, 1965), p. 235.

The validity of this method has been established using finite element analysis. The method has also been used to determine the yield strength of several materials for which the indentation loading curves are available in the literature.

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