Experimental verification and theoretical analysis of the relationships between hardness, elastic modulus, and the work of indentation

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(Received 24 April 2008; accepted 21 May 2008; published online 11 June 2008)

The relationship between hardness (H), reduced modulus (E_r), unloading work (W_u), and total work (W_t) of indentation is examined in detail experimentally and theoretically. Experimental study verifies the approximate linear relationship. Theoretical analysis confirms it. Furthermore, the solutions to the conical indentation in elastic-perfectly plastic solid, including elastic work (W_e), H, W_t, and W_u are obtained using Johnson's expanding cavity model and Lamé solution. Consequently, it is found that the W_e should be distinguished from W_u, rather than their equivalence as suggested in ISO14577, and $(H/E_r)/(W_u/W_t)$ depends mainly on the conical angle, which are also verified with numerical simulations. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2944138]

Instrumented indentation has become a powerful tool for measuring mechanical properties of surface layers of bulk materials and thin films in the past two decades. Recently ISO 14577 (Ref. 1) and ASTM E 2546-07 (Ref. 2) were published. In these two standards, indentation modulus and hardness are determined using initial unloading slope, contact area, and peak load by the method of Oliver and Pharr.[3] The method, however, depends on estimating contact area under load, which is sometimes difficult, especially when "piling up" occurs. On the other hand, ISO 14577 also defines the plastic and elastic parts of the indentation work: during the removal of the test force the remaining part is set free as work of the elastic reverse deformation $W_{\text{elast}}(\int_{h_p}^{h_{\max}} F dh)$, and $W_{\text{total}}(\int_0^{h_{\max}} F dh) = W_{\text{elast}} + W_{\text{plast}}$, where $h_{\max}$ and h_p are the indentation depth at $F_{\max}$ and the permanent indentation depth after removal of the test force, respectively. In 1998, another method was suggested by Cheng and Cheng,[4] based on the approximate linear relationship between H/E_r and W_u/W_t using dimensional analysis and finite element calculations. This method has drawn many attentions in recent years[5–7] for it is considered to be independent of material sinking in and piling up. However, the works of Alkorta *et al.*[8] and Malzbender[9] indicated that this method also faces the same limitations as the method of Oliver and Pharr and can bring significant error for soft materials.

In this letter, first, twenty sets of data from numerous indentation experiments accumulated on our laboratory in the past seven years are chosen to reveal the approximate linear relationship between H/E_r and W_u/W_t, as in Fig. 1. The experiments were carried out using a modified Berkovich indenter (equivalent half angle of conical indenter $\alpha = 70.3°$) with different depths from 1 to 3 μm. Then the questions (1) whether the unloading work is equivalent to the elastic work and (2) which parameter dominates this ratio between H/E_r and W_u/W_t lead us to solve this problem analytically. Expanding cavity model (ECM) developed by Johnson[10] is used to get the analytical solution of hardness, elastic work, and total work, and Lamé solution[11] used to get unloading work.

Considering a three-dimensional, rigid, conical indenter with a given half angle, α, indents normally into a homogeneous elastic-perfectly plastic solid, with material properties as E (Young's modulus), ν (Poisson's ratio), and Y (yield stress). Johnson's ECM assumes the process of indentation as spherical shell expansion by a hydrostatic pressure p in a hemispherical "core" of radius a (Fig. 2), which be considered as contact radius also. The elastic-plastic boundary lies at a radius c, where $c > a$. The stress and displacement outside the core are radial symmetry, which leaves only $u(r)$ for displacement component. Further, this leads to all the components ε_{ij} and σ_{ij} with $i \neq j$ zero, $\varepsilon_\theta = \varepsilon_\varphi$, and $\sigma_\theta = \sigma_\varphi$, where r, θ, and φ denote the coordinates in the spherical coordinate system.

By solving this problem, the displacement, the stress, and strain fields in the elastic region ($r \geq c$) are expressed as

$$u = \frac{(1+\nu)Y}{3E}\frac{c^3}{r^2}, \tag{1}$$

$$\varepsilon_r = -\frac{2(1+\nu)Y}{3E}\left(\frac{c}{r}\right)^3 = -2\varepsilon_\theta, \tag{2}$$

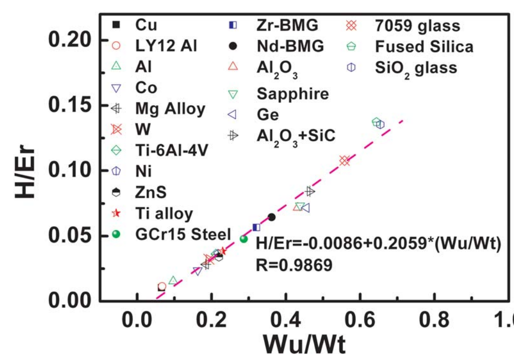

FIG. 1. (Color online) Relationship between H/E_r and W_u/W_t indicated by 20 sets of experimental data with fitting line (dash curve).

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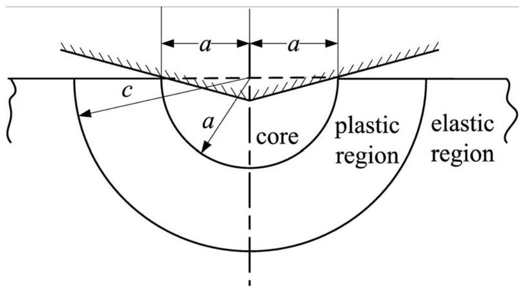

FIG. 2. A schematic illustration of stress field with hydrostatic core radius a and plastic region outer radius c for conical indentation.

$$\sigma_r = -\frac{2Y}{3}\left(\frac{c}{r}\right)^3 = -2\sigma_\theta. \tag{3}$$

Thus the energy densities for elastic part and total in the elastic region, w_e^e and w_t^e, are given by

$$w_t^e(r) = w_e^e(r) = \int \sigma_{ij} d\varepsilon_{ij} = \frac{(1+\nu)Y^2}{3E}\left(\frac{c}{r}\right)^6. \tag{4}$$

Those for the plastic region ($a \leqslant r \leqslant c$) are

$$u = \frac{-2(1-2\nu)Y}{3E}\left(r + 3r\ln\frac{c}{r}\right) + \frac{(1-\nu)Y}{E}\frac{c^3}{r^2}, \tag{5}$$

$$\varepsilon_r = -\frac{2(1-2\nu)Y}{3E}\left(3\ln\frac{c}{r} - 2\right) - \frac{2(1-\nu)Y}{E}\left(\frac{c}{r}\right)^3, \tag{6}$$

$$\varepsilon_\theta = \varepsilon_\varphi = -\frac{2(1-2\nu)Y}{3E}\left(3\ln\frac{c}{r} + 1\right) + \frac{(1-\nu)Y}{E}\left(\frac{c}{r}\right)^3, \tag{7}$$

$$\sigma_r = \left(-2\ln\frac{c}{r} - \frac{2}{3}\right)Y, \tag{8}$$

$$\sigma_\theta = \sigma_\varphi = \left(-2\ln\frac{c}{r} + \frac{1}{3}\right)Y. \tag{9}$$

Moreover, the energy densities for elastic part and total in the plastic region w_e^p and w_t^p reduce to

$$w_e^p(r) = \frac{Y^2}{E}\left[\frac{1+\nu}{3} + 6(1-2\nu)\ln^2\frac{c}{r}\right], \tag{10}$$

$$w_t^p(r) = \frac{Y^2}{E}\left[2(1-\nu)\left(\frac{c}{r}\right)^3 + 6(1-2\nu)\ln^2\frac{c}{r} + \frac{7\nu-5}{3}\right]. \tag{11}$$

There is only a hydrostatic pressure p and the material is treated as incompressible fluid, hence, no work is done to the core region. Using Eq. (8), the pressure (also considered as hardness) is given by

$$p = H = -\sigma_r|_{r=a} = \left(2\ln\frac{c}{a} + \frac{2}{3}\right)Y. \tag{12}$$

The volume conservation of hemispherical core and $da/dc = a/c$ for conical indenter leads to

$$\frac{c}{a} = \sqrt[3]{\frac{\frac{E}{Y}\text{ctg}\,\alpha + 4(1-2\nu)}{6(1-\nu)}}. \tag{13}$$

Integrate the energy densities of plastic region and elastic region separately, insert Eq. (13), and take the master terms in W_e and W_t, the elastic work and the total work done by the indenter during loading are given by

$$W_e \approx \frac{1}{3}\frac{2\pi a^3}{3}Y\,\text{ctg}\,\alpha, \tag{14}$$

$$W_t \approx \frac{2\pi a^3}{3}(Y\,\text{ctg}\,\alpha)\ln\frac{c}{a}. \tag{15}$$

When unloading, the pressure of core region reduces to zero, by subtracting the elastic stress distribution (the familiar Lamé solution[11]) from the stress distribution of loading, the residual stress field can be obtained. The superposed elastic stress components are (for both elastic and plastic regions)

$$\sigma_r^* = p\frac{a^3}{r^3}, \tag{16}$$

$$\sigma_\theta^* = \sigma_\varphi^* = -p\frac{a^3}{2r^3}. \tag{17}$$

Integrating this elastic field leads to the unloading work, which can be written as

$$W_u = \frac{\pi(1+\nu)}{2E}p^2a^3, \tag{18}$$

thus unloading work to total work can be expressed as

$$\frac{W_u}{W_t} = \left[3 + \frac{1}{\ln(c/a)}\right]\frac{(1+\nu)}{2\,\text{ctg}\,\alpha}\frac{H}{E} \approx \frac{3(1+\nu)}{2\,\text{ctg}\,\alpha}\frac{H}{E}. \tag{19}$$

Term $\ln(c/a)$ is omitted because for metals, $Y/E \approx 0.001$ and $\nu \approx 0.3$, this value reduces to about 1.5. Applying the definition of reduced modulus $E_r = E/(1-\nu^2)$ leads to

$$\frac{H}{E_r}\Big/\frac{W_u}{W_t} = \frac{2(1-\nu)}{3}\text{ctg}\,\alpha. \tag{20}$$

We can see that the ratio of H/E_r to W_u/W_t mainly depends on α which is the only geometry parameter of conical indenter. Cheng *et al.*[12] gave the fitting relationship of this value in Eq. (16) of their paper, which gives as

$$\frac{H}{E_r} = \kappa\frac{W_u}{W_t}, \tag{21}$$

where $\kappa = 1/[\lambda(1+\gamma)]$ for $60° \leqslant \theta \leqslant 80°$. Inserting λ and γ from equations of their paper, we compare our value in Eq. (20) with κ in Table I, together with our finite element cal-

TABLE I. Comparison of ratios obtained from analytical solution, FEM calculations, and fitting relation of Cheng *et al.* for $\nu = 0.3$.

α (deg)	Analytical	FEM	Cheng *et al.*
60	0.269	0.266	0.269
70.3	0.167	0.171	0.174
80	0.082	0.091	0.089

culations result using ABAQUS.[13]

Based on the work of Sneddon,[14] the ratio for linear elastic material (whose $W_u/W_t=1$) is given by

$$\frac{H}{E_r}\bigg/\frac{W_u}{W_t}=\frac{H}{E_r}=\frac{\text{ctg }\alpha}{2}, \tag{22}$$

noting the force applied on a conical indenter when the contact radius is a equals $(\pi E_r a^2 \text{ ctg } \alpha)/2$. This ratio of H/E_r to W_u/W_t depends only on α. When taking $\nu=0.25$, Eq. (20) can reduce to Eq. (22).

However, the result of Eq. (20) is only valid when plastic region radius is greater than hydrostatic core radius, that is $c/a>1$, and Eq. (13) reduces to

$$\frac{Y}{E}<\frac{\text{ctg }\alpha}{2(1+\nu)}. \tag{23}$$

When taking $\alpha=70.3°$, the upper limit of Y/E leads to 0.119 if $\nu=0.5$. Most materials may agree with the range of this limit.

By examining Eqs. (14) and (18), it is easy to find that W_u is different from W_e. Taking one of our finite element method (FEM) calculations which has been carried out with the case of $Y/E=0.005$ ($E=200$ GPa), $\nu=0.3$ and indentation depth of 100 μm for example, we obtained W_e from ALLSE (strain energy for whole model) and W_u from ALLWK (external work), it is found that $W_e=3.59$ mJ while $W_u=2.53$ mJ. The definition in ISO 14577-1(2002), Fig. A.5, in which the two areas that the unloading curve divides the area under loading curve into are indicated as W_{elast} and W_{plast}, is misleading. According to the results of present paper, the unloading work and the elastic part of total work do not equal to each other except in particular cases such as perfectly elastic and perfectly rigid plastic. Due to residual stress, W_u is always less than W_e, thus to take the area under the unloading curve to be the total elastic work W_{elast} is not proper. For the same reason, the other area should not be called plastic work because there is still partial elastic work left after unloading.

In summary, it is found that unloading work is a part of elastic one, not the total of elastic one as ISO 14577 defines. The ratio of H/E_r to W_u/W_t depends mainly on the geometry of indenter, which manifests the physical essence for the energy approaches of indentation and makes these approaches reasonable. In the future, we will systematically study the cases for more common materials such as that with work hardening.

The authors would like to thank Dr. Yang-Tse Cheng, Professor Che-Min Cheng, Professor Zhong Ling, Jing Zhang, Yihui Feng, Qiang Fu, Xuwei Wang, and Xin Jiang for their kind help. We acknowledge the support of the NSFC (10432050, 10572142, and 10721202).

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