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An energy-balance analysis for the size effect in low-load hardness testing

JIANGHONG GONG*, YING LI
Department of Materials Science and Engineering, Tsinghua University, Beijing 100084, People's Republic of China
E-mail: gong@tonghua.com.cn

The size effect in low-load hardness testing is analyzed theoretically using an energy-balance approach. A new semi-empirical equation is proposed to correlate the hardness test load and the resulting indentation size. The validity of this new equation is verified by analyzing the previously reported experimental data. It is found that the value of true hardness of material estimated with this new equation is independent of the indentor geometry as well as indentation size. © *2000 Kluwer Academic Publishers*

1. Introduction

Resistance to permanent deformation, especially deformation by indentation, is usually described with indentation hardness, H, which is traditionally defined as the ratio of the applied load, P (N), to the resulting indentation area, A (mm^2), i.e.:

$$H = \frac{P}{A} = \frac{\kappa P}{d^2} \tag{1}$$

where d is the measured length of indentation diagonal and κ is a constant equal to 1.8544 for Vickers hardness testing and to 14.229 for Knoop hardness testing.

The indentor gives geometrically similar indentations, so that it follows that the measured hardness must be independent of the applied load. However, it is experimentally well established that, more frequently, the apparent hardness measured in a low load range increases with decreasing load [1–5], and this effect is known as the indentation size effect (ISE). The existence of ISE implies that, if hardness is used as a material selection criterion, it is clearly insufficient to quote a single hardness number.

The origin of the ISE is still a controversial subject. Several possible explanations have been proposed. The most common explanations found in the literature are experimental errors related to the smallness of the indentation [6–8]. The second set of explanations are related to the intrinsic structural factors of the test materials [9–11]. Detailed reviews of the research efforts on the ISE can be found elsewhere [12–14].

The ISE has been traditionally described through the application of the Meyer's law [15], in which the applied load, P, and the resulting indentation size, d, is correlated as:

$$P = Ad^n \tag{2}$$

where A and n are descriptive parameters derived from the curve fitting of the experimental results. The Meyer exponent n has been experimentally observed to be between 1 and 2 [4]. Although Meyer's law has been well-proved suitable for representing the experimental data, an explanation of the physical meaning of this relationship has not been satisfactorily achieved.

Frohlich *et al.* [16] have proposed another empirical equation to correlate the indentation load and the resulting indentation size:

$$P = \sum_i a_i d^i \tag{3}$$

where i is a series of integers. By limiting the number of terms and assuming a_0 is zero for $P = 0$, one gets:

$$P = a_1 d + a_2 d^2 \tag{4}$$

Equation 4 is of the same form as has been applied by Bernhardt [17] when studying the ISE for several materials. Basing on the consideration of energy-balance, Bernhardt [17] suggested that the first term of the right side of Equation 4 represent the surface energy contribution while the second term represent the volume energy contribution. According to Equation 4, the total load, P, is now separated into two parts, and only the second term of the right side of Equation 4 is related to the permanent deformation by indentation. Thus a load-independent hardness, sometimes referred to as the true hardness, H_T, can be defined as [18–20]:

$$H_T = \frac{(P - a_1 d)}{A} = \kappa\left(\frac{a_2 d^2}{d^2}\right) = \kappa a_2 \tag{5}$$

The experimental basis of the energy-balance explanations for Equation 4 is the fact that, when the experimental results are represented on a $P/d \sim d$ plot, a straight line is always obtained [2, 5, 18–20]. However, recent works by the present authors [21, 22] have shown that the linear relationship between P/d and d may only

* Author to whom all correspondence should be addressed.

be held in a narrower range of applied loads. When a relatively wider range of applied loads is considered, or when a more accurate data-treatment process is used, Equation 4 is found to be insufficient. A modification of Equation 4 is also proposed in our recent works, i.e. [21, 22]:

$$P = c_0 + c_1 d + c_2 d^2 \tag{6}$$

where c_0, c_1, and c_2 are constants.

The objective of the present work is to reexamine the size effect in low-load hardness testing based on an energy-balance consideration and thence to give some theoretical foundation to Equation 6, the empirical equation proposed previously.

2. Theoretical consideration

Equation 1 may be transformed as:

$$Pd = \frac{Hd^3}{\kappa} = \beta H d^3 \tag{7}$$

Note that the indentation size, d, is directly proportional to the indentation depth, h, if the experimental errors related to the smallness of the indentation are neglected. One can consider the left side of Equation 7 as a measure of the work done by the applied load during indentation and the right side as a measure of the energy used to produce the permanent deformation. Therefore, Equation 7 is an energy-balance equation essentially, i.e., the indentation hardness is defined originally based on an energy-balance consideration. Exactly, hardness is a parameter with a dimension of J/m^3, rather than N/m^2 or GPa, which is a measure of the energy needed for producing the permanent deformation of an unit volume.

In continuation to this idea, it is clear from Equation 7 that a constant value of hardness may be expected if the total work done by the applied load is transformed to energy for the permanent deformation without any extra dissipation. Unfortunately, the extra energy-dissipation is always inevitable during indentation, for a part of the work done by the applied load must be transformed to the energy for the increase of the surface area of specimen due to indentation. The extra energy-dissipation may also result from a variety of phenomena, including the formation and propagation of microcracks, the formation of pile-ups near the indentation, the migration of the grain boundaries, the deformation of the intrinsic pores, etc. To a first approximation, the energy dissipated for all of these phenomena may be considered to be area-related and directly proportional to d^2. Thus, Equation 7 should be revised as:

$$Pd = \alpha d^2 + \beta H_T d^3 \tag{8}$$

where α is a constant dependent on the surface energy of test material and H_T is the true hardness, or indentation size-independent hardness, defined as the energy needed for the permanent deformation of an unit volume. Obviously, one can get an equation of the same form as Equation 4 by dividing both sides of Equation 8 by d.

The analysis above is similar to those reported originally by Bernhardt [17] and used afterwards by other authors [2, 5, 19, 20]. Although self-consistent conclusions have been obtained in these earlier studies by analyzing experimental results according to Equation 8 or Equation 4 [2, 5, 18, 20], it should be pointed out that Equation 8 cannot, in fact, be used directly to describe the experimental data, for the experimental errors, which may result from the elastic recovery of indentation [6], the optical resolution of the objective lens used [7], and/or the sensitivity of the load cell [8], etc., are neglected in deducing this equation. Using Equation 8 to study the ISE for fused silica, Hirao and Tomozawa [2] have shown that attributing the α term to the surface energy may yield unacceptably large surface-energy values, exceeding 0.1 J/cm^2. One possible explanation is, as suggested by Hirao and Tomozawa themselves, that the origin of the variation in surface area, including external surface area and inner surface area, is so complicated during indentation that the relationship between the surface energy and the measured α-value cannot be determined more accurately. As can be seen later, however, errors due to estimating α-value directly from the originally measured data with Equation 8, without considering the experimental errors, may be another important cause for the resulting large values of surface energy.

Now let us incorporate the experimental errors, which are usually inevitable in the conventional hardness testing, into Equation 8. Firstly, we denote the true values of the applied load and the resulting indentation size as P_0 and d_0, respectively. Clearly, it is these two quantities that can be correlated by Equation 8, i.e.,

$$P_0 = \alpha d_0 + \beta H_T d_0^2 \tag{8a}$$

In general, the experimental errors in hardness testing can be divided into two sets: one related to the measurement of indentation size and the other to the measurement of applied load. Several methods have been proposed for correcting these experimental errors [23–25]. The simplest and commonly used method is to modify the measured results by a constant "error", i.e., the true values of the applied load, P_0, and the resulting indentation size, d_0, can be obtained with [25]:

$$\begin{cases} P_0 = P + \eta \\ d_0 = d + \delta \end{cases} \tag{9}$$

where P and d are now the measured data, and η and δ are constants dependent on test material, test machine, and test conditions.

Substituting Equation 9 into Equation 8a yields

$$\begin{aligned} P = {} & \left(\beta H_T \delta^2 + \alpha\delta - \eta\right) + (2\beta H_T \delta + \alpha) d \\ & + \beta H_T d^2 \end{aligned} \tag{10}$$

Equation 10 is of the same form as the empirical equation established previously by the present author

[21, 22], Equation 6. Thus the physical meanings of the parameters in Equation 6 can be understood by the following equations:

$$c_0 = \beta H_T \delta^2 + \alpha\delta - \eta \tag{11a}$$

$$c_1 = 2\beta H_T \delta + \alpha \tag{11b}$$

$$c_2 = \beta H_T \tag{11c}$$

i.e., all of the parameters in Equation 6, c_0, c_1, and c_2, are functions of the true hardness, surface energy, and the experimental errors. Note that the c_2-value is dependent only on the true hardness of test material. Equation 11c can be used as the basic equation for estimating the true hardness, H_T, the energy needed for producing the permanent deformation of an unit volume.

Up to now, a reasonable explanation can be offered for size effect in low-load hardness testing. Substituting Equation 10 into Equation 1 yields:

$$H = \kappa\left(\frac{c_0 + c_1 d + c_2 d^2}{d^2}\right) = \kappa\left(\frac{c_0}{d^2} + \frac{c_1}{d}\right) + H_T \tag{12}$$

or

$$\Delta H = H - H_T = \kappa\left(\frac{c_0}{d^2} + \frac{c_1}{d}\right) \tag{12a}$$

Equation 12a reveals that a difference, ΔH, exists between the apparent hardness defined as Equation 1, H, and the true hardness, H_T. Because ΔH in Equation 12a decreases with increasing d in most cases, the ISE, i.e., a decreasing tendency in apparent hardness with increasing indentation size, is observed more frequently. On the other hand, it is clear that an increasing tendency in ΔH with increasing d may also be expected from Equation 12a. So an opposite or reverse form of the indentation size effect, namely RISE, in which the apparent hardness increases with increasing indentation size, may also be predicted. In fact, such a phenomenon has been observed, although rarely, in the previous studies by Banerjee and Feltham [26, 27]. The fact that a constant value is always obtained for the apparent hardness when measurement is conducted at a relatively higher level of applied load can also be explained with Equation 12a. The absolute value of ΔH decreases with increasing identation size, hence with the applied load. When the applied load increases to a limited level, namely P_c, the value of ΔH would become so small that it can be neglected compared with the value of true hardness, H_T. Thus the apparent hardness can keep nearly constant.

3. Experimental verifications

The conclusions obtained in the theoretical consideration mentioned above are now examined using the experimental data published previously by Sakai *et al.* [28]. For convenience, the values of the apparent hardness and the true hardness will be presented with a dimension of GPa in the following analysis, although it has been demonstrated in the preceding section that the hardness is a parameter with the dimension of J/m^3.

A stoichiometric translucent mullite ($3Al_2O_3 \cdot 2SiO_2$) was used in [28]. Specimens were divided into three sets and then tested as-received (denoted as M-AR), after annealed at 1750 °C for 5 h (M-75), or after annealed at 1800 °C for 5 h (M-80), respectively. Both Vickers hardness and Knoop hardness were measured for all of the three sets of specimens. The original indentation data were listed in Table III and IV of [28] and now are reproduced in Figs 1–3 where the abscissa is the indentation size, d, the average length of the two diagonals for Vickers indentation or the length of the major diagonal for Knoop indentation, and the coordinate is the applied load, P. The solid lines in these plots are obtained by a conventional polynomial regression according to Equation 6. In each case, the regression analysis returns a correlation coefficient of 0.999 or better. Clearly, Equation 6 is proven sufficiently suit-

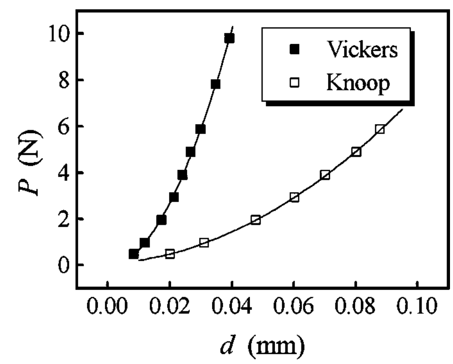

Figure 1 Plot of applied load as a function of indentation size for sample M-AR.

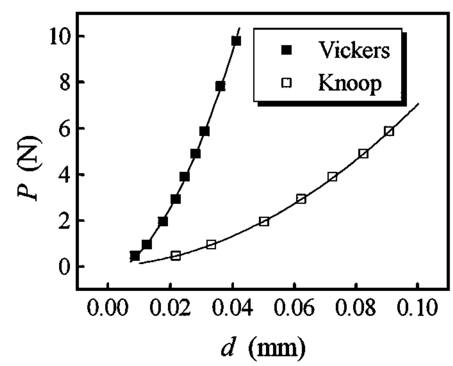

Figure 2 Plot of applied load as a function of indentation size for sample M-75.

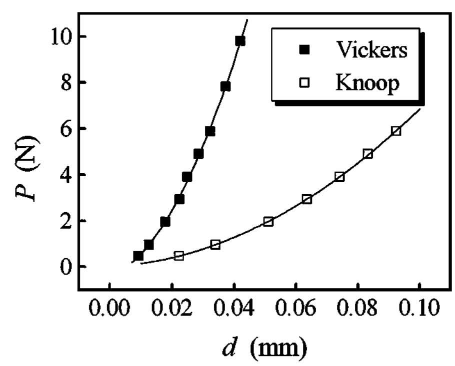

Figure 3 Plot of applied load as a function of indentation size for sample M-80.

TABLE I Best-fit values of parameters in Equation 6

Sample	Vickers indentation				Knoop indentation			
	c_0 (J/m)	c_1 ($\times 10^3$ J/m^2)	c_2 ($\times 10^6$ J/m^3)	H_{TV} (GPa)	c_0 (J/m)	c_1 ($\times 10^3$ J/m^2)	c_2 ($\times 10^6$ J/m^3)	H_{TK} (GPa)
M-AR	−0.210	30.97	5792.70	10.7	0.057	9.33	644.99	9.2
M-75	−0.407	56.30	4763.43	8.8	0.013	8.43	622.19	8.9
M-80	−0.395	53.77	4527.83	8.4	0.022	7.41	608.80	8.7

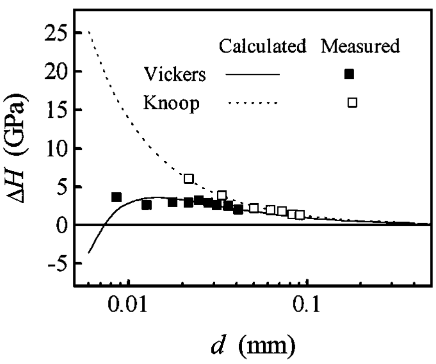

Figure 4 Indentation size-dependence of the apparent hardness for sample M-75.

able for the representation of the experimental data. The best-fit values of the parameters included in Equation 6 for the three sets of specimens are recorded in Table I.

According to the theoretical consideration, the true hardness of test material, H_T, can be calculated conveniently from the best-fit value of parameter c_2 in Equation 6 with Equation 11c. The calculated values of the true Vickers hardness, H_{TV}, and the true Knoop hardness, H_{TK}, are also given in Table I. It is clearly evident that, for each sample, the values of true hardnesses obtained from two indentor geometries, H_{TV} and H_{TK}, are in good agreement with each other, giving a convincing support for the present theoretical consideration.

Using the best-fit values of parameters c_0 and c_1 listed in Table I, the differences between the values of the apparent hardness and the true hardness, $\Delta H_V = H_V - H_{TV}$ and $\Delta H_K = H_K - H_{TK}$, are calculated as functions of indentation size for sample M-75 according to Equation 12a and then illustrated in Fig. 4. Also shown in Fig. 4 are the measured results of those two quantities. Fig. 4 indicates that the apparent Vickers hardness is predicted to increase sharply at first and then decrease slowly with increasing indentation size, while the apparent Knoop hardness decreases continuously in the range of indentation size considered. A constant value of the apparent hardness can be expected when the indentation size, d, is larger than about 0.5 mm for both Vickers and Knoop hardness testings.

A further comment should be made on the experimental results presented in Fig. 4. As shown in Fig. 4, the apparent Vickers hardness is nearly constant in the indentation size range of 8–40 μm. It should be pointed out, however, that such a phenomenon is simply an artifact and cannot be considered the same as observed usually in a relatively higher level of applied load, for the variation of ΔH_V with d in this range of indentation size is too small to be detected accurately. As predicted with Equation 12a, the solid line in Fig. 4, the indentation size-dependence of the apparent Vickers hardness will be observed easily if a wider range of the applied load is used.

For purposes of comparison, the original indentation data shown in Figs 1–3 are analyzed according to Equation 4. Equation 4 in the alternative form is:

$$\frac{P}{d} = a_1 + a_2 d \qquad (13)$$

It can be expected from Equation 13 that a plot of P/d vs. d should yield a straight line. Table II summarizes all of the a_1 and a_2 values obtained by linear regression according to Equation 13, as well as the values of load-independent hardness, H_0, which is calculated with Equation 5, for M-AR, M-75, and M-80. It is shown that, the load-independent hardness obtained with Vickers indentation, H_{V0}, is significantly higher than that obtained with Knoop indentation, H_{K0}, indicating that Equation 4 does not give an accurate explanation for the ISE.

In fact, the P/d-d relationships for series of Si_3N_4-based ceramics have been found to be significantly non-linear, although a correlation coefficient of 0.99 or better is usually returned for these analyses [21, 22]. Similar conclusion can also be obtained by analyzing the original indentation data for millite. Fig. 5a represents the Vickers indentation data of sample M-75 on a P/d-d plot. The solid line in this plot represents "best-fit" of Equation 13 to the measured data. It can be seen clearly that all the data fall into two separate parts, both showing good linearity. The fact that a good

TABLE II Best-fit values of parameters in Equation 4

Sample	Vickers indentation			Knoop indentation		
	a_1 (N/mm)	a_2 (N/mm^2)	H_{V0} (GPa)	a_1 (N/mm)	a_2 (N/mm^2)	H_{K0} (GPa)
M-AR	7.47	6323.63	11.7	12.18	616.94	8.8
M-75	11.62	5743.36	10.7	9.48	616.03	8.8
M-80	10.98	5448.67	10.1	8.58	697.38	8.5

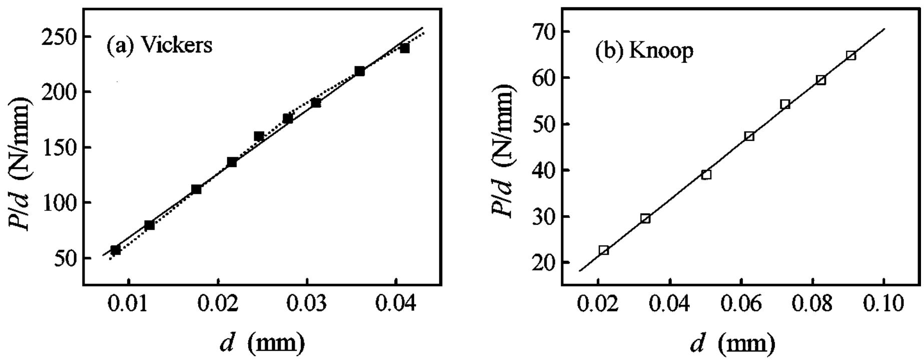

Figure 5 Plots of P/d as a function of d for sample M-75 (a) Vickers indentation; (b) Knoop indentation.

linear relationship between P/d and d is observed with the Knoop indentation data of sample M-75, Fig. 5b, may be an artifact due to the extremely small value of c_0, 0.01 J/m.

The final comment concerns experimental results reported by Hirao and Tomozawa [2], which has been mentioned in the preceding section. Similarly, it is reasonable to assume that the original Knoop indentation data for fused silica can be described with Equation 6 in which the value of parameter c_0 is determined to be zero, for an excellent linearity between P/d and d has been obtained with those data [2]. Note that the value of parameter c_1 in Equation 6 is dependent on both experimental error, δ, and surface energy, α, as predicted in Equation 11b. It can be concluded easily that neglecting the experimental errors is an important cause for the resulting large values of surface energy.

4. Concluding remarks

The essence of the analysis presented in this study is that hardness is a parameter which is defined essentially based on the energy-balance consideration. The origin of the indentation size effect on the apparent hardness lies in the fact that the traditional definition for hardness, the ratio of the applied load to the resulting indentation area, is an uncompleted description for the energy-balance relationship during indentation. Based on a detailed theoretical analysis, the following energy-balance equation is proposed to correlate the hardness test load, P, and the resulting indentation size, d:

$$P = c_0 + c_1 d + c_2 d^2$$

where the constants c_0, c_1, and c_2 are functions of true hardness, surface energy, and the possible experimental errors. Especially, the c_2-value is dependent only on the true hardness of test material.

The validity of this new equation is then examined using the previously published experimental data. It is shown that this equation gives excellent fits to the experimental results. In addition, the true hardness estimated with this equation is found to be independent of indentor geometry as well as indentation size.

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