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Empirical method for the determination of hardness from low-load ball indentation tests for brittle materials

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Ball indentation test has been established as a powerful means for measuring hardness for metals and alloys for a long time and the outputs of this test are known as the Brinell hardness. In order to yield a reliable hardness number, it is generally required that the resultant indentation impression should be large enough, i.e., the ratio of the radius of the resultant plastic impression, a, to the indenter radius, R, should be larger than 0.25 [1]. Little effort has been devoted to the determination of hardness of brittle glasses and ceramics with ball indentation. This may be attributed to the facts that: (1) brittle glasses and ceramics are usually harder than metals and alloys, making it very difficult to produce an indentation impression with an a/R ratio larger than 0.25 at low load levels, and (2) when a hard sphere is pressed against the brittle materials under a sufficiently high load, the resultant contact stress would cause a preexisting flaw in the specimen surface to propagate, resulting in some uncertainties in the hardness determination.

On the other hand, the microcracking behavior of brittle materials associated with ball indentation has been studied extensively [2] and theoretical analysis and experimental observations have confirmed that the minimum applied load necessary to propagate the preexisting surface flaws can be used to determine the fracture toughness of the test material [3]. Furthermore, the ball indentation experiments have also been applied to study the surface residual stresses [4], the surface flaw densities [5] and the local strength [6]. In these previous studies, significant permanent deformation was usually observed in brittle materials when being indented with hard spheres, even if the resultant indentation impression has an a/R ratio smaller than 0.25. Some experimental results previously reported for 9 mol% Ce-TZP ceramics [7] are shown in Fig. 1, where the ordinate is the "indentation stress," $p_0 = P/\pi a^2$ and the abscissa is the "indentation strain," a/R. Also shown in Fig. 1 is a solid line predicted for the sample TZP-III by the classical Hertzian relation for purely elastic contacts [2],

$$p_0 = \left(\frac{3E}{4\pi k}\right)\frac{a}{R} \tag{1}$$

where k is a dimensionless constant determined by the Young's modulus, E, the Poisson's ratio, υ, of the test material and the Young's modulus, E_I, the Poisson's ratio, υ_I, of the indenter,

$$k = \frac{9}{16}\left[(1-\upsilon^2) + \left(1-\upsilon_\mathrm{I}^2\right)\frac{E}{E_\mathrm{I}}\right] \tag{2}$$

The derivation of the measured data from the ideal elastic behavior indicates that significant permanent deformation would occur even at the lowest indentation load. Similar phenomena were also observed for other brittle materials [8].

In this letter, we will show that an empirical method for determining the hardness of brittle materials may be established by analyzing the indentation stress-strain curves, such as those shown in Fig. 1, measured with ball indentation.

We start our analysis by defining the hardness as the ratio of the work done by indentation load, W, to the permanent deformation zone volume, V. Such a definition was frequently adopted in the previous studies [9, 10] concerning the analysis of the indentation size effect, i.e., an experimental phenomenon that the measured hardness decreases or increases with increasing indentation load. For brittle materials, the ball indentation stress-strain curve measured in low load regime can be approximately treated to be linear. Thus, to the first approximation, we have

$$W = \frac{1}{2}Ph \tag{3}$$

where $h = R - (R^2 - a^2)^{0.5}$ is the indenter penetration depth at peak load.

From geometry, the permanent deformation zone volume, V, can be expressed as

$$V = \frac{\pi}{6}h_\mathrm{p}\left(3a^2 + h_\mathrm{p}^2\right) \tag{4}$$

where h_p is the final depth of the resultant indentation impression measured after full unloading.

Using Equations 3 and 4, we obtain the basic equation for hardness calculation

$$H = \frac{W}{V} = \frac{3Ph}{\pi h_\mathrm{p}\left(3a^2 + h_\mathrm{p}^2\right)} \tag{5}$$

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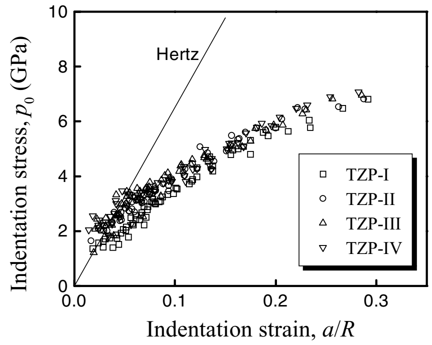

Figure 1 Indentation stress versus indentation strain plots for some Ce-TZP ceramics. Indentation tests were conducted using WC balls of radius $R = 1.2$–12.5 mm over a load range $P = 200$–3000 N. See ref. [7] for details about the materials and the test procedures.

In general, $3a^2 \gg h_p^2$. Thus, Equation 5 can be approximated as

$$H = p_0\left(\frac{h}{h_p}\right) \tag{6}$$

Equation 6 implies that the hardness, H would be equal to the indentation stress or the mean contact stress, p_0, if $h_p = h$. However, nanoindentation tests conducted with ball or pyramid indenters [11, 12] have confirmed that the final depth of the contact impression measured after full unloading is usually smaller than the indenter penetration depth, implying that elastic recovery occurs during the unloading half-cycle. This is to say that, at least for indentation impressions made under small load, H would be larger than p_0 because $h_p < h$. Noting that hardness should be a material constant, one can infer from Equation 6 that p_0 would increase with increasing h_p/h.

It is usually difficult to measure h_p during conventional Brinell indentation tests. Therefore, Equation 6 cannot be directly used to calculate hardness number. However, an empirical method for extracting the hardness number from the indentation stress-strain curves such as those shown in Fig. 1 can be proposed based on the above analysis. Re-plotting the experimental data shown in Fig. 1 as Fig. 2, where the indentation stress is plotted as functions of h/R. As can be seen, if the h/R ratio is selected as the measure of the indentation strain, the stress-strain curves exhibit an interesting feature: p_0 increases gradually with h/R and a plateau value of p_0 can be expected when h/R tends to be infinite. One can expect that the elastic recovery may be so small that it can be neglected, i.e., $h_p/h \approx 1$, when the indentation is made under a very high load. Thus, it is reasonable to infer that the plateau value of p_0 shown in Fig. 2 can be treated, at least approximately, to be the hardness number of the test material.

Now we try to determine the plateau value of p_0. This can be done easily by noting the similarity between the stress-strain curves shown in Fig. 2 and the typical cyclic fatigue deformation curves. In low-amplitude cyclic fatigue tests conducted on metals [13] and single crystalline silicon [14], it was generally observed that, as the cumulative shear-strain, γ, increases, the maximum stress in each cycle, σ, increases gradually and then tends to a saturation value when γ tends to infinity. Gong *et al.* [15] proposed the following equation to describe the cyclic fatigue deformation curve measured on single crystalline silicon,

$$\sigma = \sigma_\infty - \sigma_G \exp\left(-\frac{\gamma}{\lambda_G}\right) - \sigma_M \exp\left(-\frac{\gamma}{\lambda_M}\right) \tag{7}$$

where the parameters σ_∞, σ_G, σ_M, λ_G and λ_M are adjustable constants.

Gong *et al.* [15] suggested that the second and the third terms in the right-hand side of Equation 7 represent the effects of the dislocation generation and the dislocation motion on the cyclic deformation resistance, respectively. Because the material resistances to dislocation generation and dislocation motion increase as the cumulative strain increases, the values of both the second and the third terms would decrease as γ increases and become zero when a well-developed dislocation structure forms. Undoubtedly, the deformation behavior of a material during indentation is somewhat similar to that during cyclic fatigue. Thus, an expression similar to Equation 7 may be employed to describe the indentation stress-strain curves, i.e., we can write

$$p_0 = H - p_G \exp\left(-\frac{h/R}{\lambda_G}\right) - p_M \exp\left(-\frac{h/R}{\lambda_M}\right) \tag{8}$$

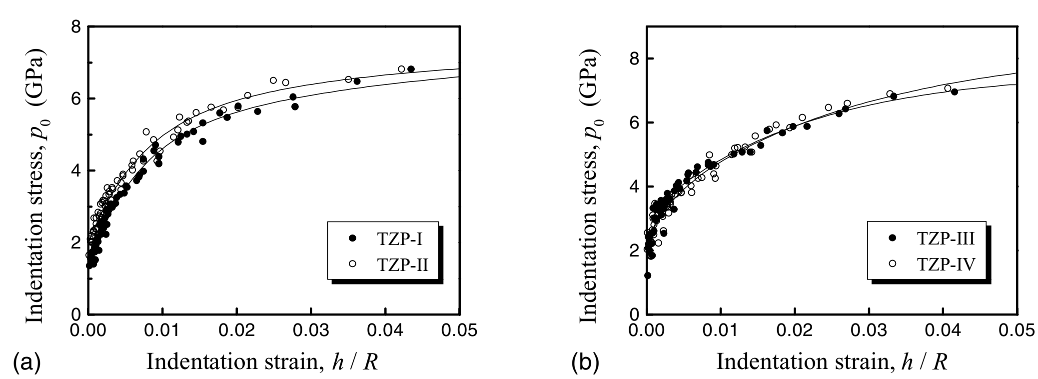

Figure 2 Replotting the experimental data shown in Fig. 1 in the p_0 versus h/R scale.

TABLE I Comparison between the hardness values determined from different methods

Material	TZP-I	TZP-II	TZP-III	TZP-IV
Hardness from this study (GPa)	7.30	7.31	7.61	8.41
Vickers hardness [7] (GPa)	6.85	7.28	7.28	7.11

where the parameters H, p_G, p_M, λ_G and λ_M are adjustable constants. Following the above analysis, H in Equation 8 is the plateau value of p_0, i.e., the hardness number we want to determine.

The solid lines shown in Fig. 2 are obtained by analyzing the experimental data according to Equation 8. It is evident that, for each material, Equation 8 provides a satisfactory description for the experimental data. The best-fit value of H for each material is compared with the reported Vickers hardness in Table I. Considering that the Vickers hardness was determined using only one load level (100 N) [7] and there may be some uncertainties in the Vickers hardness data due mainly to the indentation size effect, there is reason to believe that the good agreement between these two sets of data is evident, giving a sound support for our analysis.

In summary, we established an empirical method for extracting a hardness number from the indentation stress-strain curves measured from low-load ball indentation tests. This method can be used for the hardness measurements for brittle materials, which are usually so hard and brittle that conventional Brinell hardness measurements cannot be performed.

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