

## Letter to the Editor

**Comment on “Measurement of hardness on traditional ceramics”, H. Kim and T. Kim, J. Eur. Ceram. Soc., 22, 1437–1445 (2002)**

Indentation hardness testing is a convenient means of investigating the mechanical properties of a small volume of materials. However, the existence of so-called indentation size effect (ISE), i.e. the experimental phenomenon that the measured hardness increases as the applied indentation load decreases, makes it insufficient to quote a hardness number measured at a single load level for material characterization and impossible to compare directly the hardness data between different materials.<sup>1</sup> To explain the origin of the ISE in ceramics, two approaches were proposed, one being the proportional specimen resistance (PSR) model<sup>2</sup> and the other based on the energy balance consideration.<sup>3</sup> In these two models, the relationship between the applied indentation load,  $P$ , and the resultant indentation size,  $d$ , was predicted to be

$$\frac{P}{d} = a_1 + a_2d \quad (1)$$

where  $a_1$  is a constant and  $a_2$  is suggested to be related to the so-called *true hardness*,  $H_0$ ,<sup>2,3</sup>

$$H_0 = ka_2 \quad (2)$$

where  $k$  is a constant. For Vickers indentation,  $k = 1.8544$ .

Recently, Gong et al. re-examined the applicability of Eq. (1) to the indentation size effect observed in ceramics. It was found that Eq. (1) is insufficient for describing the experimental data. By considering the effects of the machining-induced plastically deformed surface<sup>4,5</sup> and the experimental errors related to the smallness of the indentation on the hardness measurements,<sup>6,7</sup> Eq. (1) was modified as

$$P = a_0 + a_1d + a_2d^2 \quad (3)$$

According to the previous analyses,<sup>4–7</sup> the physical meaning of the parameter  $a_2$  in Eq. 3 is the same as that in Eq. (1) and  $a_0$  and  $a_1$  are constants.

Recently, Kim and Kim<sup>8</sup> performed indentation hardness testing on some traditional ceramics, roofing

tiles, and observed significant ISEs in all the six samples they considered. When the indentation data for a given sample were analyzed according to Eq. (3), they obtained a negative value of  $a_2$  and then suggested that the parameter  $a_2$  in Eq. (3) cannot be used as an index of the so-called *true hardness*. This conclusion conflicts with those deduced in the previous studies,<sup>4–7,9–11</sup> in which the experimental data obtained on many typical materials were confirmed to obey Eq. (3). In this communication, a possible explanation for such a confliction was presented.

The first point needed to be emphasized here is that both Eqs. (1) and (3) were proposed based on the analyses of the ISE observed in single crystals or polycrystalline ceramics with high relative densities, usually larger than 95%. On the other hand, all the six samples examined by Kim and Kim<sup>8</sup> exhibit very low relative densities, ranging from 88 to 56%. There have been several papers<sup>12–14</sup> concerning the effect of porosity on the measurement of the *apparent* hardness, i.e. the hardness defined by the ratio of the applied indentation load to the area of the resultant indentation impression. It was generally reported that the measured apparent hardness decreases with the increasing porosity, or the decreasing relative density. However, little effort has been devoted to the effect of porosity on the determination of the so-called *true hardness* from the existing models such as PSR model, Eq. (1), and its revised version, Eq. (3). Therefore, a simple analysis should be conducted here to incorporate the effect of porosity into the relationship between the indentation load and the resultant indentation size.

We start from the energy-balance model for the ISE, which was proposed firstly by Fröhlich et al.<sup>15</sup> and then developed by Quinn and Quinn for ceramics.<sup>3</sup> The energy-balance model can be understood easily by analyzing the equation which is traditionally used to define the apparent hardness of material:

$$H = k \frac{P}{d^2} \quad (4)$$

where  $k$  is a constant dependent on the indenter geometry, the same parameter as that appeared which in Eq. (2). Rewriting Eq. (4) as:

$$Pd = Hd^3/k \quad (5)$$

The left side of Eq. (5) can be considered as a measure of the work done by the applied load during an indentation event, while the right side of Eq. (5) is the energy used for producing the permanent deformation. Clearly, Eq. (5) is an energy-balance equation and one can conclude that the apparent hardness is defined originally based on the assumption that, during indentation, the total work done by the applied load transforms to energy for the permanent deformation without any extra dissipation. However, it should be pointed out that, during indentation, the extra energy dissipation is always inevitable. The extra energy dissipation may occur due to the increases in surface area, the formation of microcracks, and other mechanisms.<sup>3</sup> To a first approximation, the extra energy dissipation may be treated to be area-related and directly proportional to  $d^2$ . Thus, Eq. (5) should be revised as:

$$Pd = \alpha d^2 + H_T d^3/k \quad (6)$$

where  $\alpha$  is a constant and  $H^1$  is the true hardness. Dividing both sides of Eq. (6) by  $d^2$  yields Eq. (1).

The essential prerequisite for deducing Eq. 6 is that the values of the applied load and the resulting indentation size can be measured accurately. However, this prerequisite cannot be satisfied easily in experiments. Due to the optical resolution of the objective lens and/or the sensitivity of the load cell, experimental errors in the measured indentation size and the measured test load can be expected to exist for any indentation testing. In addition, the elastic recovery of the indentation may also result in a value of indentation size smaller than the actual one. Therefore, it is clear that Eq. (6) cannot be used directly to describe the original data measured with a conventional hardness testing. It can be used only when the experimental errors mentioned above are considered properly. Based on this consideration, Eq. (6) was further modified to the form of Eq. (3),<sup>6,7</sup> in which the effect of experimental errors in indentation load and indentation size are considered properly in the parameters  $a_0$  and  $a_1$  and the parameter  $a_2$  is proven to be independent of the experimental errors.

Note that, in the analysis mentioned earlier, the change in the area of the free surfaces of the test material was assumed to be proportional to  $d^2$  and has little effect on the parameter  $a_2$ . However, we will show below that this assumption is not tenable if the changes in the shape of pores due to indentation are considered.

Considering a material with a total porosity of  $p$ . For the sake of simplification, the pores in the material were assumed to be spherical in shape with the same radius of  $r$ . During indentation, the pores in the prospective indentation zone would be compressed, resulting in a

change in free surface area,  $\Delta S_p$ . Assuming the deformed pores to be a circle in shape, we obtain

$$\Delta S_p = N(4\pi r^2 - \pi r'^2) = 3N\pi r^2 \quad (7)$$

where  $N$  is the number of the pores in the prospective indentation zone and can be determined from

$$p = \frac{V_p}{V_0} = \frac{N(4\pi r^3/3)}{d^2 h/6} \propto 2N \left(\frac{r}{d}\right)^3 \quad (8)$$

where  $p$  is the porosity,  $V_p$ , is the total volume of the pores in the prospective indentation zone before indentation and  $V_0$  and  $h$  are the volume and the depth of the indentation-induced impression ( $h \propto d$ ).

Thus we can deduce from Eqs. (7) and (8) that  $\Delta S_p$  is proportional to  $d^3$ , rather than  $d^2$ , i.e.

$$\Delta S_p = 3N\pi r^2 \propto p \left(\frac{d^3}{r}\right) \quad (9)$$

In continuation of the earlier analysis, one can conclude that the energy-balance model should be further modified to incorporate the effect of porosity. Ignoring the effect of experimental errors, the modified form of the energy-balance model can be written as

$$Pd = (\alpha d^2 - \beta \Delta S_p) + \gamma d^3 \quad (10)$$

where  $\alpha$ ,  $\beta$  and  $\gamma$  are constants. The first term in the right hand side represents the contribution from the change in free surfaces due to the formation of new indentation surfaces and microcracking and the second term from the changes in the shapes of the pores. The minus in the parentheses is introduced because the changes in the shapes of the pores would result in a decrease in the area of the free surfaces. Inserting Eq. (9) into Eq. (10) gives

$$Pd = \alpha d^2 + \left(-\frac{\beta' p}{r} + \gamma\right) d^3 \quad (11)$$

where  $\beta'$  is another constant.

Note that  $p = N_0(4\pi r^3/3) \propto r^3$  (where  $N_0$  is the number of pores in a unit volume), i.e.  $r \propto p^{1/3}$ . Thus one can deduce that the ratio of  $p/r$  is proportional to  $p^{1/3}$  and obtain

$$Pd = \alpha d^2 + (-\beta'' p^{2/3} + \gamma) d^3 \quad (12)$$

Comparing Eq. (12) with Eqs. (1) or (3) gives

$$a_2 = -\beta'' p^{2/3} + \gamma \quad (13)$$

Eq. (13) shows that porosity plays an important role in the determination of the true hardness by analyzing

the experimental data based on the energy-balance model. There is reason to believe that a similar conclusion may also be obtained for the PSR model, although the corresponding analysis may be more complex.

Fig. 1 shows the variation of  $p^{2/3}$  with  $p$ . As can be seen, the value of  $p^{2/3}$  varies slightly in the range of  $p < 1\%$  and sharp changes in  $p^{2/3}$  due to the change in  $p$  can be observed only in the high porosity region. According to Eq. (13), the tendency shown in Fig. 1 implies that a nearly porosity-independent  $a_2$ -value would be observed in the low porosity region while  $a_2$  would decrease significantly as the porosity increases within the high- $p$  region. Undoubtedly, a negative  $a_2$ -value would be obtained if the porosity of the test material is high enough. This seems to be a possible explanation for the facts that positive  $a_2$ -values were generally reported for materials with high relative densities<sup>4–7,9–11</sup> and negative  $a_2$ -values were observed by Kim and Kim<sup>8</sup> who examined materials with low relative density.

In the energy-balance model, the parameter  $a_2$ , the index of the true hardness, was defined based on the energy needed for producing the permanent deformation of a unit volume.<sup>3,6,7,15</sup> In the previous studies, the effect of pores on the energy-balance relationship was ignored and  $a_2$  was related only to the energy used for the plastic deformation due to indentation. Considering the existence of pores, permanent deformation due to indentation now includes two components, one being the plastic deformation and the other being the densification of the pores in the prospective indentation deformation zone. The decrease in the area of the free surfaces of the pores due to densification may release extra energy, thereby reducing the resistance to permanent deformation. As a result,  $a_2$  would decrease. For materials with high porosities, the energy provided by the densification of pores would be high enough for producing the plastic deformation, i.e.,  $\beta'' p^{2/3} > \gamma$  in Eq.

(13), thus a negative  $a_2$ -value would be expected. In this case, the work done by the applied indentation load is transformed to energy for producing new indentation surfaces, rather than plastic deformation. Therefore, one can conclude that the occurrence of a negative  $a_2$ -value is reasonable when analyzing the indentation data according to Eq. (3).

It should be pointed out that the analysis conducted in the preceding section is somewhat simplified and phenomenological. In fact, the parameter  $\beta''$  in Eq. (13) depends strongly on the statistical distributions of the shapes and the sizes of pores. Such a dependency may be very complex due to the intrinsic microstructural inhomogeneity of the test material and may vary from material to material. Therefore, a direct comparison between the  $a_2$ -values obtained with Eq. (13) for materials with different porosities may be questioned. In other words, it should be very careful to use the parameter  $a_2$  defined in Eq. (13) as an index of true hardness, especially for materials with high porosities. Fortunately, as can be predicted from Fig. 1, the first term in the right hand side of Eq. (13) varies slightly within the low-porosity region and has a value close to zero, implying that Eq. 13 can be used approximately to determine the true hardness for materials with low porosity,  $p < 1\%$ . In this case,  $a_2$  equals approximately to  $\gamma$ , the energy needed for producing the permanent plastic deformation of a unit volume during indentation.

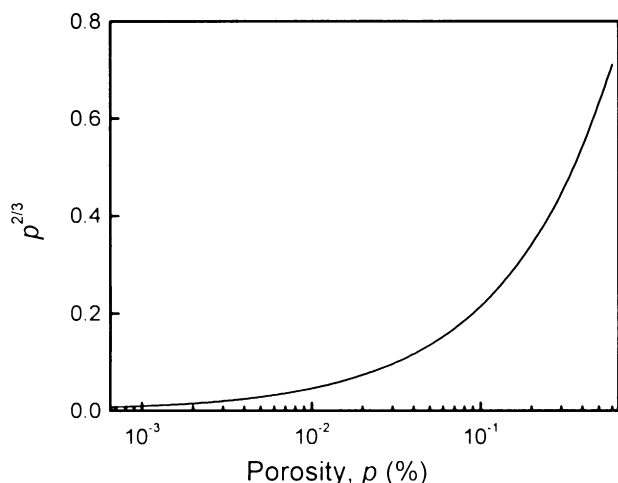


Fig. 1. Variation of  $p^{2/3}$  with  $p$ .

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