Analysis of the indentation size effect on the hardness of alumina ceramics using different models

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Received 6 June 2008, received in revised form 2 March 2009, accepted 13 March 2009

Abstract

The Vickers hardness of slip-cast high purity alumina ceramics is determined as a function of the indentation load. The results show that the measured hardness depends crucially on the load, which indicates the influence of the indentation size effect (ISE). The load dependence of hardness is analysed by using the traditional Meyer's law, a proportional specimen resistance (PSR) model and a modified proportional specimen resistance (MPSR) model. The best correlation between measured values and used models is achieved by using a modified PSR model.

K e y w o r d s: hardness, indentation size effect, alumina ceramics

1. Introduction

Hardness is one of the most frequently measured properties of ceramics and it is a measure of material resistance to permanent plastic deformation. Its value helps to characterize the resistance to deformation, densification and fracture. Hardness is usually measured on conventional microhardness machines with Vickers or Knoop diamond indenters. These machines make impressions whose diagonal size is measured with an attached optical microscope. For research purposes, Vickers, Knoop and Berkovich (triangular pyramid) indenters are customary; Rockwell and Brinell indenters are rarely suitable for ceramics research. For engineering and characterization applications, approximately 60 % of worldwide published ceramic hardness values are Vickers hardness values, with loads typically in the range of a few N to 9.8 N (HV 1) and occasional data for soft or high-toughness ceramics as high as 98 N (HV 10). About 35 % are Knoop hardness values with loads from as low as 0.98 N (HV 0.1) to 19.6 N (HV 2). At low indentation loads, problems arise from the load dependence on hardness and from the measurement uncertainty due to small indentation size. At higher loads, cracking and spalling pose problems; in some cases they make credible measurement impossible.

Accurate measurements of the hardness of brittle materials have been difficult due to the dependence of hardness on load. For ceramics, the indentation size is small and the percentage error may be larger; because of that it is crucial that the diagonal length be measured carefully. Diagonal lengths produced in ceramics are smaller than in most metals for a given load, which increases the measurement uncertainty.

A correct optical microscopy technique is crucial for ceramic hardness measurements. Reasonable skill, experience and a careful experimental technique are necessary to measure diagonal lengths accurately and precisely.

The indenter gives a geometrically similar indentation so that the measured hardness should be independent of the applied load. But investigations have confirmed that the hardness is usually load-dependent, i.e. decreasing in a low load range [1–8] or increasing [9] with an increase in the applied load. Some materials exhibit an increase in hardness with a decreasing load (indentation size effect, ISE) while others undergo a decrease in hardness with a decreasing load (reverse indentation size effect, RISE) [1, 7]. The ISE is illustrated schematically in Fig. 1.

Apparent hardness is a function of the applied load at low indentation test loads, where there is no constant value for hardness (load dependent hardness).

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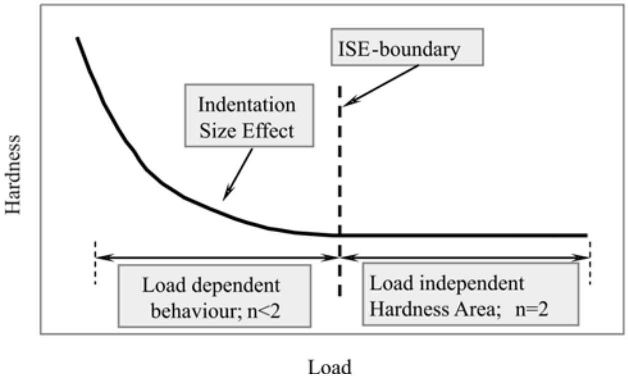

Fig. 1. Schematic plot of the ISE behaviour [9].

Table 1. Average values of indents and standard deviation of indents as a function of the applied load

Load (kg)	Average indent size (μm)	Standard deviation
0.05	5.7	0.23
0.1	8.6	0.33
0.2	13.3	0.43
0.5	21.9	0.78
1	34.0	0.91
3	61.2	1.47
5	78.6	1.7

At high indentation test loads, hardness is a constant value. Load independent hardness has also been referred to as the "true" hardness in some literature.

To calculate the ISE, several relationships between the applied load, F, and the resulting indentation size, d, have been suggested [1–9]. The goal of this work is to analyse the observed ISE of slip-cast alumina ceramics using the traditional Meyer's law, a proportional specimen resistance (PSR) model and a modified PSR model.

2. Experimental procedure

The material used in this study was a high purity slip-cast alumina ceramics (SC-Al_2O_3). An SC-Al_2O_3 specimen was prepared in Applied Ceramics, Inc., Fremont, California, U.S.A.

Vickers hardness measurements HV 0.05, HV 0.1, HV 0.2, HV 0.5, HV 1, HV 3, and HV 5 were performed using indentation loads of 0.4903 N, 0.9807 N, 1.961 N, 4.903 N, 9.807 N, 29.43 N, and 49.03 N for 15 s, respectively. Indentation tests were carried out under laboratory conditions on an Instron, Wilson-Wolpert Tukon 2100B, a micro Vickers tester (HV 0.05 – HV 1) and Vickers tester Zwick (HV 3 and HV 5). Care was taken to make indentations only on those areas, which had no visible pores. Thirty measurements for each load were performed on the alumina sample. The average value of diagonal lengths of the indentation for each load was used to calculate the hardness according to Eq. (2). Before carrying out hardness measurements, all specimens were prepared by the standard ceramographic technique [10]. The sample was mounted with a DuroFix-2 Kit, ground and then polished up to 1 μm with a diamond paste until a mirror-like surface was achieved. All indentation tests were carried out under ambient laboratory conditions.

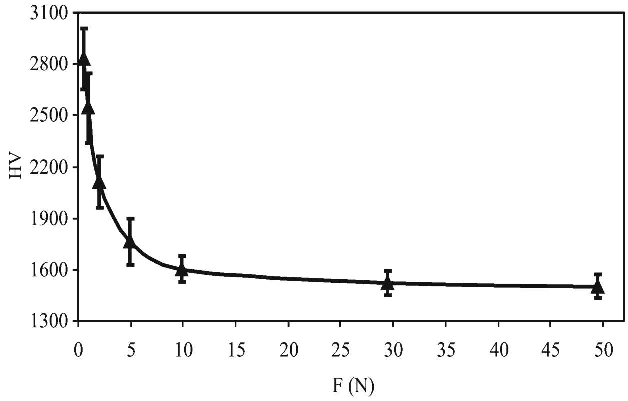

Fig. 2. Vickers hardness as a function of the applied load for SC-Al_2O_3 ceramics (mean value and standard deviation).

3. Results and discussion

The hardness number H is defined as a ratio of the applied load F to the contact area A between the indenter and the sample:

$$H = F/A. \tag{1}$$

In the case of Vickers hardness tester, hardness numbers HV were calculated using the relation:

$$\mathrm{HV} = F/A = \alpha F/d^2, \tag{2}$$

where $\alpha = 0.1891$, HV is Vickers hardness, F is applied load (N), d is arithmetic mean of the two diagonals (mm).

Results of average values of indents and standard deviation of indents as a function of the applied load are shown in Table 1. The results show that the indent level increases with the increasing indentation test load.

The variation of hardness of SC-Al_2O_3 ceramics with the applied load is shown in Fig. 2. All data presented in Fig. 2 are averages of thirty values; therefore error bars are plotted. Error bars characterize the accuracy.

Table 2. Regression analysis results of experimental data according to Eq. (3)

Sample	n	$\log A$	A (N mm^{-n})	Correlation factor R^2
SC-Al_2O_3	1.7461 ± 0.023	3.5917 ± 0.039	3906	0.9991

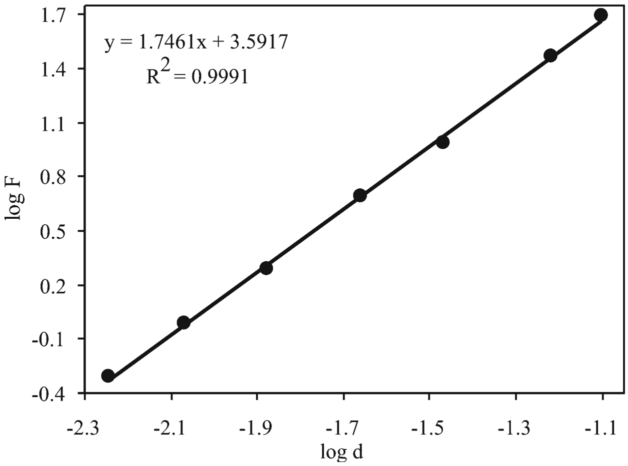

Fig. 3. Vickers hardness data on SC-Al_2O_3 ceramics according to the Meyer's law.

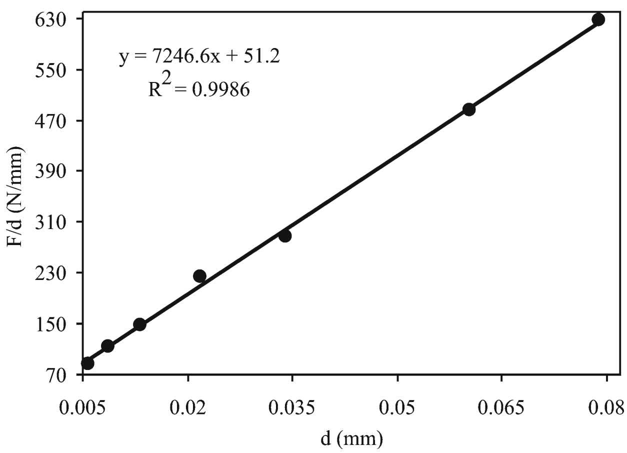

Fig. 4. Vickers hardness data on SC-Al_2O_3 ceramics according to the PSR model.

Results show that the hardness decreases with the increasing indentation test load. This dependence of hardness on the applied load is known as the ISE. The observed decrease in hardness with an increasing load is usually called a normal ISE.

The most common explanation for the ISE found in literature is directly related to the structural factor of the test material. Meyer proposed an empirical relation between the load and the size of the indentation. This relationship is usually called the Meyer's law [5]. The Meyer's law is simply an empirical expression to describe the relationship between the indentation load F and the resultant indentation size d:

$$F = Ad^n, \tag{3}$$

where A is a constant parameter for a given material and n is Meyer's index (or number), which is a measure of the ISE. These parameters are obtained from the curve fitting (Fig. 3) of experimental results of indentations ($\log F$ versus $\log d$). Meyer's law has been suitable for the representation of experimental data for a variety of ceramics, and Meyer's index n has been experimentally observed to be between 1.5 and 2.0. The A and n values in Eq. (3) were determined by linear regression analysis and the results are summarized in Table 2. The data show a linear relationship, indicating that the traditional Meyer's law is suitable for describing microindentation data. The Meyer's index n obtained experimentally is less than 2, which indicates that hardness is dependent on test loads.

The Meyer's law is simply an empirical expression to describe the relationship between the indentation load and the indentation size. An alternative analysis of the ISE to the Meyer's law is a PSR model and recently a number of researchers have explained the ISE with this model. The PSR model was introduced by Li and Bradt [4]. In this model, the applied test load F and the resulting indentation size d were found to follow the relationship:

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

where a_1 and a_2 were considered as constants; a_1 (N mm^{-1}) coefficient was suggested to relate to the proportional resistance of the test specimen and a_2 (N mm^{-2}) to the load-independent hardness, or "true" hardness. Equation (4) can be transformed into:

$$F/d = a_1 + a_2 d. \tag{5}$$

The parameters a_1 and a_2 from Eq. (5) are evaluated through the linear regression of F/d versus d. Figure 4 shows the plot F/d versus d with a straight line with a slope equal to the a_2 value and an intercept equal to the a_1 value. The results are presented in Table 3. Li et al. [11] concluded that this model might provide a satisfactory explanation for the origin of the ISE in hardness tests for different materials.

Gong et al. [5] suggested a modified PSR model

Table 3. Regression analysis results of experimental data according to a PSR model

Sample	a_1 (N mm^{-1})	a_2 (N mm^{-2})	Correlation factor R^2
SC-Al_2O_3	51.2 ± 5.1	7246.6 ± 123.2	0.9986

Table 4. Parameters F_0, a_1 and a_2 of the MPRS model according to Eq. (5) for SC-Al_2O_3 ceramics

Sample	F_0 (N)	a_1 (N mm^{-1})	a_2 (N mm^{-2})	Correlation factor R^2
SC-Al_2O_3	0.1839 ± 0.229	34.408 ± 16.078	7454.2 ± 189.5	0.9999

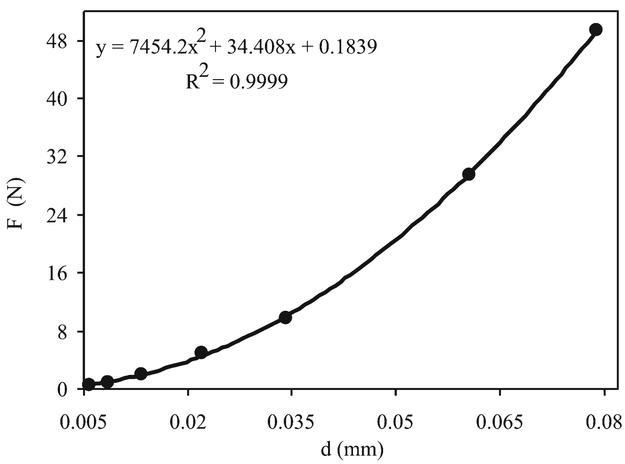

Fig. 5. The applied load versus indentation size according to the modified PSR model for SC-Al_2O_3 ceramics.

designed on the basis of the consideration of the effect of a machining-induced, residually stressed surface on the hardness measurements, giving

$$F = F_0 + a_1 d + a_2 d^2, \tag{6}$$

where F_0 is a constant related to the surface residual stresses associated with the surface machining and polishing and a_1 and a_2 are the same parameters as the ones given in Eq. (4). Figure 5 illustrates F versus d for alumina ceramics samples tested in the present study. The solid line in this plot is obtained by conventional polynomial regression according to Eq. (6). The best-fit values of the parameters included in Eq. (6) for SC-Al_2O_3 ceramics sample are presented in Table 4.

F_0 is a specimen constant, rather than a material constant. Surface finishing processes (grinding and polishing), which remove the material mechanically, used in the preparation of the specimens prior to indentation can be a cause of the ISE because the finishing processes introduce plastic deformation and cracks into the material adjacent to the surface [5]. The relatively small values of F_0 in Table 4 seem to be reasonable estimates of the residual of the surface stress for SC-Al_2O_3 ceramics, which have been subjected to careful finishing processes (grinding and polishing).

4. Conclusions

Like many other ceramics, SC-Al_2O_3 ceramics show the indentation size effect behaviour. It means that the measured hardness values increase with decreasing indentation loads.

Meyer's law has proved to be satisfactory for the description of experimental data with a correlation coefficient of 0.9991. The Meyer's index n obtained experimentally is less than 2, which indicates that hardness is dependent on test loads.

Moreover, the PSR model can be used to analyse the ISE observed in SC-Al_2O_3 ceramics, but the best correlation between measured values and mathematical models was achieved with the MPSR model with a correlation coefficient of 0.9999.

Acknowledgements

The presented research results were achieved within the scientific project Structure and properties of engineering ceramics and ceramic coatings supported by the Croatian Ministry of Science, Education and Sport. We thank Matt Sertic from Applied Ceramics, Inc. for providing alumina ceramics samples.

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