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INDENTATION SIZE EFFECT IN THE VICKERS HARDNESS FOR ALUMINA CERAMICS

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ABSTRACT

Objective of this study is to determine the effect of indentation load on the Vickers hardness of cold isostatically pressed (CIP) high purity alumina (Al_2O_3) ceramics. Specimen of a CIP-Al_2O_3 ceramics was embedded in DuroFix-2 Kit, polished, then indented at loads ranging between 0.1, 0.2 and 0.5 kg with a Vickers hardness tester (n=30 per load). This study show that conventional hardness measurements are load-dependent, which implies a substantial influence of the indentation size effect (ISE). The ISE was described through the application of the Mayer Law, a proportional specimen resistance (PSR) model and a modified PSR model.

Keywords: hardness, indentation size effect, alumina ceramics.

1. INTRODUCTION

Indentation hardness testing is a convenient means of investigating the mechanical properties of a small volume materials. Hardness characterizes the ceramic`s resistance to deformation, densification, displacement and fracture. Although measuring and interpretating ceramic hardness should be routine, pitfalls, controversies and surprises abound. In generaly, measurements show that the hardness of material tends to increase with decreasing load. The load dependence of the hardness is refered to indentation size effect (ISF). The ISE has been studied for severel decades. In recent years, there has been renewed interest in the indentation size effect (ISE) in brittle materials. The ISE has been attributed to number of fenomena/mechanisms, including: the ratio of elastic/plastic deformation during the formation of the impression and any subsequent effect of elastic recovery, surface dislocation pinning, work hardening during indentation, the optical resolution of the objective lens used, the effect of machining-induced residually stressed surface on the hardness measurement, etc.[1-9]. Despite all these mechanisms being considered, the cause of ISE is still a topic of debate.

2. MATERIALS AND METHODS

The material used in the study was a cold isostatically pressed (CIP)-Al_2O_3 with purity of 99.8 %. Al_2O_3 ceramic contains MgO as sintering aid and usual impurities SiO_2, CaO, Na_2O and Fe_2O_3. The CIP-Al_2O_3 specimens were supplied by Applied Ceramics, Inc., Fremont, California, U.S.

Vickers hardness measurements HV0.1, HV0.2 and HV0.5 were performed using indentation loads: 0.9807 N (0.1 kg), 1.961 N (0.2 kg) and 4.903 N (0.5 kg) for 15 s, respectively. Indentation tests were carried under laboratory conditions on Instron, Wilson-Wolpert Tukon 2100B, micro Vickers tester. Care is taken to make indentations only on those areas which have no visible pores. Thirty determinations for each load were performed on alumina sample. The average value of the diagonal lengths of the indentation for each load was used to calculate the hardness according equation (1). Before hardness measurements all specimens were prepared by the standard ceramographic technique [10]. The sample was mounted with DuroFix-2 Kit, grinded and than polished up to 1 μm with a diamond paste until a mirror-like surface was achieved. All the indentation tests were carried under ambient laboratory conditions.

The ISE reported here was described quantitatively through the application of the Mayer Law, a proportional specimen resistance (PSR) model and a modified PSR model [1-9] to best correlate measured values with mathematical models.

3. RESULTS AND DISCUSSION

The values of the Vickers hardness were calculated, by the following equation:

$$HV = P/A = \alpha F/d^2 \qquad (1)$$

Where:
HV = Vickers hardness,
α = 1.8544
F = applied load (N),
d = arithmetic mean of the two diagonal length (m).

The Vickers hardness of alumina ceramics under different loads is shown in Figure 1. Each of the data points represents an average of measurements from thirty tests at each load. Obtained results indicate that the load dependence of the hardness is evident for alumina ceramics.

ISE is traditionally described on the basis of Meyer`s Law. In the order to analyze the ISE in the hardness testing it is needs to fit the experimental data according to the Meyer`s Law which correlate the applied load and the resulting indetation size:

$$P=Ad^n \qquad (2)$$

where d is the indentation diagonal length under an applied test load P. The parameter A is constant parameter for given material and n is Meyer`s index (or number), which is a measure of the ISE. The parameter n is the slope of the straight line obtained when $\log P$ is plotted against $\log d$, and A is the intercept. Figure 2 obtained by plotting $\log P$ against $\log d$ for alumina ceramics shows straight lines, with correlation coefficient of 1. The n value indicates the extent of ISE. From the curve fitting of experimental results of the indentation obtained value of n is 1.6679 and $\log A$ is 3.4534. The Meyer`s index n obtained experimentally is under 2, which indicates that hardness is dependent on test loads.

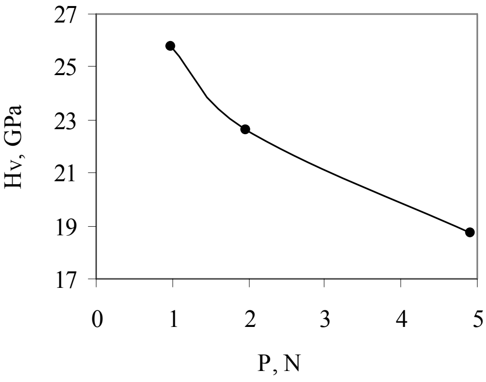

Figure 1. Vickers hardness as a function of the applied load for alumina ceramics.

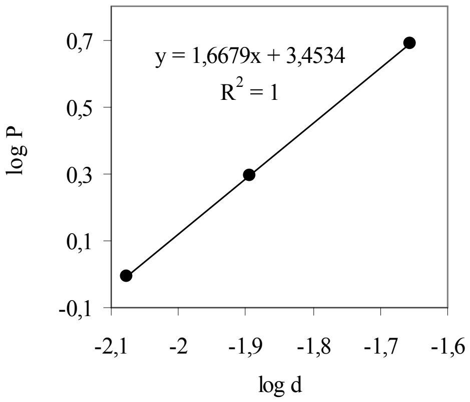

Figure 2. Vickers microhardness data onalumina ceramics according to the Meyer law.

The Meyer Low is simply an empirical expression to describe the relationship between indentation load and the results intentation size. Recently a number of researches have explained the indentation size effect with the proportional specimen resistance (PSR) model [2,7-9]. The proportional specimen resistance (PSR) model was introduced by Li and Bradt [1]. In this model, the applied test load, P, and the resulting indentation size, were found to follow the relationship:

$$P = a_1 d + a_2 d^2 \qquad (3)$$

where a_1 and a_2 were considered as constants; a_1 (N/mm) 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 (3) can be transformed into :

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

The parameters a_1 and a_2 from equation (4) are evaluated through the linear regression of P/d versus d. Figure 3 shows the plot P/d versus d with straight line with a slope aqual to the a_2 value and an intercept equal to the a_1 value. The results are presented in Table 1. Li et al. [11] concluded that this model might provide a satisfactory explanation for the origin of the ISE in microhardness tests for different kind materials.

Table 1. Regression analysis results of experimental data according to a PSR model .

Sample	a_1	a_2	Correlation factor (R^2)
CIP-Al_2O_3	42	8255.9	0.9984

Gong et al. [5] suggested a modified PSR (MPSR) model designed on the basis of the consideration of the effect of a machining-induced, residually stressed surface on the hardness measurements, giving:

$$P=P_o+a_1d+a_2d^2 \tag{5}$$

Where P_0 is a constant related to the surface residual stresses associates with the surface machining and polishing and a_1 and a_2 are the same parameters as the ones given in equation (3). Figure 4 illustrates P versus d for alumina ceramics samples tested in the pressed study. The solid line in this plot is obtained by conventional polynomial regression according to equation (5). The best-fit values of the parameters included in equation (5) for CIP-Al_2O_3 ceramics sample are presented in Table 2.

Table 2. Parameters P_0, a_1 and a_2 of the MPRS model for CIP-Al_2O_3 ceramcs.

Sample	P_0	a_1, N/mm	a_2, N/mm^2	Correlation factor (R^2)
CIP-Al_2O_3	-0.20	84.62	6661.9	1

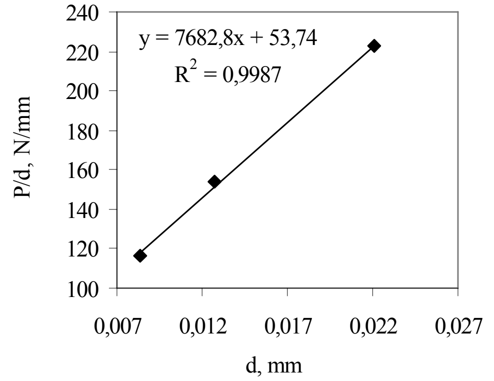

Figure 3. Vickers microhardness data on alumina ceramics according to a PSR model.

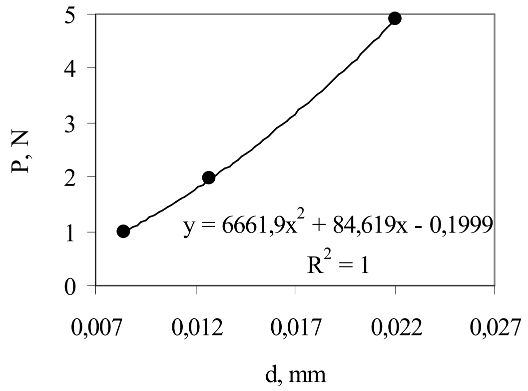

Figure 4. The applied load versus indentation size according to the modified PSR model for alumina ceramcs.

4. CONCLUSION

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

The Meyer index n is obtained experimentally (1.6679) is less than 2, which indicates that hardness is dependent on test loads.

The PSR model can be used to analyze the ISE observed in CIP-Al_2O_3 ceramics, but the best correlation between measured values and mathematical models was achieved with the Meyer Law and the modified PSR model with correlation coefficient of 1.

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