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HARDNESS AND FRACTURE TOUGHNESS OF ALUMINA CERAMICS

TVRDOĆA I LOMNA ŽILAVOST ALUMINIJ OKSIDNE KERAMIKE

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Abstract: Many methods are currently used to measure fracture toughness of ceramic materials. Methods based on crack-length measurements of cracks introduced into the sample surface by the Vicker's indentor have the advantage that they are easy to use, but they are very unreliable due to subcritical crack growth and the difficulty in determining the exact length of the cracks.
The aim of the present work is to investigate the hardness from Vickers indentations and the fracture toughness based on crack-length measurements of cracks introduced into the sample surface of cold isostatically pressed (CIP)-Al_2O_3 with 99.8 % purity.

Key words: alumina ceramics, hardness, fracture toughness

Sažetak: Puno metoda je u uporabi za određivanje lomne žilavosti keramičkih materijala. Metode bazirane na mjerenju duljina pukotina na površini uzorka nastalih Vikersovim indentorom mogu se lako primjeniti, ali su vrlo nepouzdane obzirom na podkritičan rast pukotine i teško određivanje točne duljine pukotina. Svrha ovog rada je ispitivanje tvrdoće Vikersovim indentorom i lomne žilavosti bazirane na mjerenju duljine pukotina na površini uzorka visoko čiste alumij oksidne keramike oblikovane hladnim izostatičkim prešanjem.

Ključne riječi: aluminij oksidna keramika, tvrdoća, lomna žilavost

1. INTRODUCTION

The appeal of ceramics as structural materials is based on their low density combined with their high temperature resistance, high hardness, chemical inertness and high wear resistance [1, 2]. Alumina ceramics find a wide range of applications due to its composition. Some of the major application areas can be grouped as shown in Table 1. A major goal of current ceramic research and development is to produce tough, strong ceramics that perform reliably. The fracture toughness of ceramics is still poor, compared to that of metals and composites. Fracture toughness values are used extensively to characterize the fracture resistance of ceramics and brittle materials.

Table 1. Example of applications for a range of Alumina's [3].

% Al_2O_3	Grain size	Porosity	Application Area
> 99.6	Fine	Closed	Electrical, Engineering, Biomedical
> 99.8	Fine	Zero	Lamp tubes, Optical
> 99.6 (recristallised)	Medium	Closed	High temperature uses
95 – 99.5	Fine	Closed	General electrical, Engineering
80 – 95	Fine	Closed	Low duty electrical (spark plugs)
90 – 99.6	Fine/Coarse	Open	Filter media
80 - 90	Fine/Coarse	Open	Abrasive

Due to its simplicity, its non-destructive nature, and the fact that minimal machining is required to prepare the sample, the use of the Vickers hardness indentations to measure fracture toughness (K_{IC}) has become quite popular. Indentation hardness is a measurement of the size of an indentation made by a diamond pyramid-shaped indenter of specified size and shape pressed into a polished surface by a known load. The surface is normally not etched prior to the indentation. Among a variety of indentor geometries used in hardness testing, the Vickers indentor is one in the most widespread use. The Knoop indenter has only two-fold symmetry and it is commonly used on ceramics. The Vickers indenter has four-fold symmetry but makes a deeper indentation and is more inclined to cause fractures in brittle materials than the Knoop indenter.

The Vickers diamond pyramid hardness number, *HV*, is defined as the ratio of the applied load, *F*, to the pyramidal contact area, *A*, of the indentation:

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

where *d* is the length of the diagonal of the resultant impression, and $\alpha = 0.1891$ for Vickers indentor [4-7].

Cracks associated with Vickers or Knoop hardness impressions are widely used as artificial defects of "known" size for the fracture toughness (K_{Ic}) measurement of ceramics. There are many references [5, 8-13] on the quantitative relations between the surface crack length of indentation cracks and the fracture toughness. Interest in this method stems from its simplicity and the small volume of material required to conduct K_{Ic} measurements. A Vickers indentation is implanted onto a flat ceramic surface and cracks develop around the indentation with their lengths in inverse proportion to the toughness of the material. By measuring crack lengths, it is possible to estimate K_{Ic}.

The conventional procedure of hardness testing consists of applying a fixed load on a diamond indentor and measuring, with the help of a microscopy, the dimension of the resultant indentation on the surface of the test material after unloading.

In this paper, the hardness of CIP-Al_2O_3 ceramics was measured by the Vickers indentations method (Figure 1) and cracks that originated from the Vickers indentations were used to compute the fracture toughness by the Anstis method. Toughness measured by the Anstis crack-length method is dependent on the elastic modulus of the material, microindentation hardness and crack length, and the applied load. Anstis et al. [9, 5, 13] employed a simplified two-dimensional fracture mechanics analysis and obtained:

$$K_{Ic} = 0.016 \left(\frac{E}{H}\right)^{1/2} \times \frac{F}{c^{3/2}} \tag{2}$$

where F is the load in Newtons, c is the crack length from the center of the indent to the crack tip in meters, E is the Young's modulus in GPa and H is the Vickers hardness in GPa. It must be noted that under small indentation loads, only small Palmqvist cracks form, see Figure 2. A cross-sectional view and a top view of the two most common types of cracks are shown in Figure 2. At low loads, Palmquvist cracks are favored, while at high loads fully developed median cracks result. A simple way to differentiate between the two types is to polish the surface layers away; the median crack system will always remain connected to the inverted pyramid of the indent while the Palmqvist will become detached, as shown in Figure 2 b [2, 10,12].

2. EXPERIMENTAL PROCEDURE

2.1. Sample preparation

The material used in this study was a cold isostatically pressed (CIP)-Al_2O_3 with 99.8 % purity. Chemical composition of investigated alumina ceremics is shown in Table 2. The CIP-Al_2O_3 specimens were supplied by Applied Ceramics, Inc., Fremont, California, U.S.A. Al_2O_3 ceramic contains MgO as sintering aid and the usual impurities SiO_2, CaO, Na_2O and Fe_2O_3. The specimens were rectangular coupons; size was 8 mm × 10 mm × 20 mm. The Archimed density of the CIP-Al_2O_3 was 3.91 g/cm^3, thus the CIP-Al_2O_3 ceramic specimen attained 98.09 % of its theoretical density (3.986 g/cm^3).

Table 2. Chemical composition of the Al_2O_3 ceramics.

sample	wt. %					
	MgO	Fe_2O_3	SiO_2	Na_2O	CaO	Al_2O_3
alumina ceramics	0.066	0.015	0.02	0.05	0.013	rest

2.2. The Vickers hardness and the fracture toughness testing

The Vickers hardness was measurements by means of ZWICK. All indentations were made into mirror polished surfaces. In this method, a diamond indenter is applied to the surface of the specimen to be tested. Ten Vickers impressions have been made through in the surface of the sample using load of 1 · 9.81 N (HV1) during fifteen seconds. After removal of indenter, the size of cracks that emanate (sometimes) from the edges of the indent are measured, and Vickers hardness of alumina ceramics is calculated according to equation (3). All indentation tests were carried under ambient laboratory conditions. Vickers indentation was measured by optical microscopy Olympus BH. The fracture toughness (K_{IC}) was determined by the direct crack measurement method using the Palmqvist crack model and Anstis equation (2).

3. RESULTS AND DISCUSSION

After indentation, the length of each of the two diagonals of the square-shaped Vickers indentation was immediately measured by optical microscopy with a magnification of 500. The values of the Vickers hardness were calculated, by the following equation:

$$HV1 = \frac{0.1891 \cdot F}{d^2} \tag{3}$$

Where:
HV = Vickers hardness,
F = applied load (N),
d = arithmetic mean of the two diagonal length (mm).
The arithmetic mean of the all measured diagonals length (10 impressions) was 32 µm. It means that *HV1* is 1811.

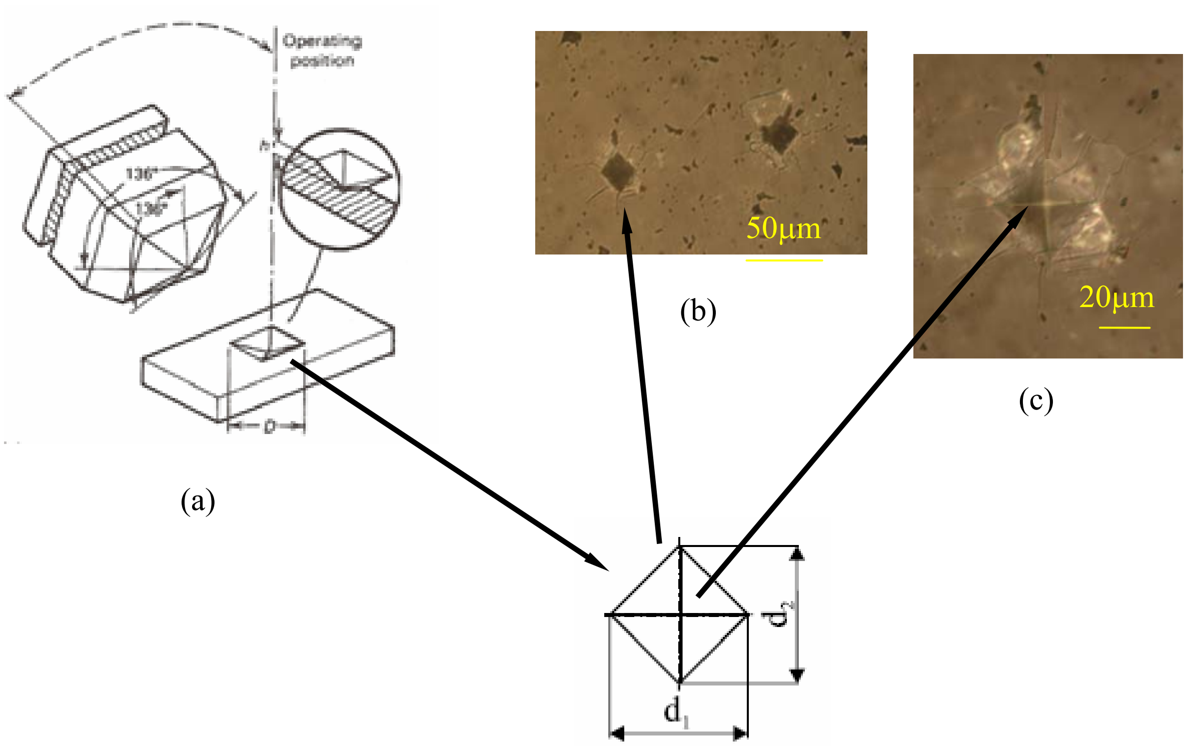

Figure 1. Vickers hardness testing. a) Schematic of a Vickers or diamond pyramid hardness indenter. The indentation depth h is approximately 1/7 of the average length *D* of the diagonals [4]. b) Vickers indentations on a radial cross section of 99.8 % Al_2O_3 ceramics. The load on the indenter was 1 · 9.81 N (HV1), and the dwell time was

15 s. c) Magnified view of the left Vickers indentation in b). Note the fracture field surrounding the indentation.

The K_{IC} values were determined using the direct crack measurement method. The Palmqvist crack model equation was used to compute the fracture toughness (K_{IC}). The Young's modulus for high purity alumina ceramics is 386 GPa. The arithmetic mean of the all measured c length (10 impressions) was 42 μm.
The calculated fracture toughness (K_{IC}) value according to equation (2) is 2.69 MPa $\sqrt{m}$.

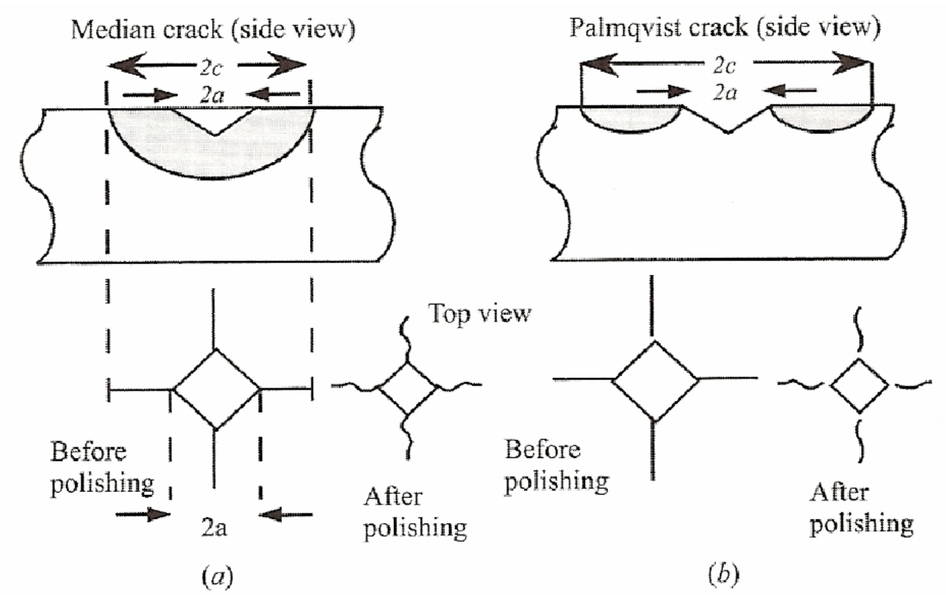

Figure 2. Crack system developed from the Vickers indents. (a) Side and top views of a median crack, (b) top and side views of a Palmqvist crack [2].

4. CONCLUSION

From the obtained results of these investigations it can be conclude that investigated method for determination of fracture toughness based on crack-length measurements of cracks introduced into the sample surface is applicable for technical ceramics such as alumina ceramics. Obtained results of hardness and fracture toughness of high purity alumina ceramics are also in conformity with results in the literature data [4].

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5. REFERENCES

[1] Reed, J.S., Principles of Ceramics Processing, John Wiley & Sons, Inc., New Yirk, 1995.
[2] Barsoum, M. W. Fundamentals of Ceramics, McGraw-Hill, 2003.

[3] http://www.azom.com
[4] R. E. Chinn, Ceramography Preparation and Analysis of Cearmic Microstructures, ASM Interantional, USA, 2002.
[5] Wachtman, J. B., Mechanical properties of ceramics, John Wiley &Sons, Inc., 1996.
[6] Musikant, S., What everyone engineering have to known about ceramics, Marcel Dekker, Inc., 1990.
[7] ISO 6507/2-Metallic materials-Hardness test-Vickers test-Part 2: HV0.2 to less lhan HV5.
[8] K. Strecker, S. Ribeiro, M. J. Hoffmann, Fracture toughness measurements of LPS-SiC: a comparison of the indentation technique and the SEVNB method, Mat. Res., **8 (2)** 2005.
[9] G.R. Anstis, P. Chantiklul, B.R. Lawn and D.B. Marshall, A critical evaluation of indentation techniques for measuring fracture toughness: I, Direct crack measurements. *J. Am. Ceram. Soc.* **64** (1981) 533–538.
[10] M.T. Laugier, Palmquist indentation crack analyses for toughness determination in WC–Co composites. *Key Eng. Mat.* **32** (1989) 77–84.
[11] K. Niihara, R. Morena and D.P.H. Hasselman, Evaluation of K_{Ic} of brittle solids by the indentation method with low crack-to-indent ratios. *J. Mat. Sci. Let.* **1** (1982) 13–16.
[12] Z. Li, A. Ghosh, A.S. Kobayashi and R.C. Bradt, Indentation fracture toughness of sintered silicon carbide in the Palmqvist crack regime. *J. Am. Ceram. Soc.* **72** (1989) 904–911.
[13] 28. D. Casellas, N. M. Nagl, e*t al.*, Growth of Surface Indentation Cracks in Alumina and Zirconia Toughened Alumina, *Key Engineering Materials*, **127-131** (1997) 895-902.