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Journal of Prosthodontic Research 57 (2013) 82–87

Journal of Prosthodontic Research

www.elsevier.com/locate/jpor

Original article

Effect of loading conditions on the fracture toughness of zirconia

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Received 1 August 2012; received in revised form 27 December 2012; accepted 22 January 2013
Available online 14 March 2013

Abstract

Purpose: A Vickers hardness indenter was pressed into yttria-stabilized zirconia (Y-TZP) by the indentation fracture method (IF method).
Methods: The effect on the calculated Vickers hardness, fracture toughness values, and indentation fracture load (9.8, 49, 98, 196, and 294 N) was examined to deduce the optimum conditions of the IF method. Calculated Vickers hardness and fracture toughness values were analyzed with one-way analysis of variance and then multiple comparisons (Scheffe). The appearance of on indentation and cracks was also evaluated using a scanning electron microscopy (SEM).
Results: Indentation of Y-TZP was generated by 9.8 and 49 N of indentation fracture load, however cracks could not be confirmed with the microscope attached to the Vickers hardness tester. Both indentation and cracks were observed at 98, 196 and 294 N of indentation fracture load obtained values of 7.1 and 6.8 $MPam^{1/2}$. Cracks noted at the 98 N were not clear, whereas the 196 and 294 N showed especially clear cracks. Due to the hardness of zirconia and the light loads, fracture toughness values for 9.8, 49, and 98 N could not be calculated. There was no significant difference between 196 and 294 N, when calculated fracture toughness values were analyzed with multiple comparisons. SEM revealed clear indentation and cracks, that extended linearly, but no chips or fractures were observed. Surface changes were observed at 196 and 294 N that are presumed to be accompanied by phase transition around the cracks.
Conclusions: Optimum experimental conditions of the indentation fracture load in the IF method were determined as 196 and 294 N.

Keywords: Zirconia; Fracture toughness; Indentation fracture method; Vickers indenter; Vickers hardness

1. Introduction

In recent dental therapy, demands for esthetics and biocompatibility of restoration devices using biomaterials are increasing [1–5]. Zirconia that is increasingly applied to a clinical setting is a biomaterial with excellent strength and toughness [6–10]. Zirconia gathers attention as a material that has functions and strength as well as, or better than, metal frames because zirconia is available for use as a substructure or framework material for the application of porcelain in the fabrication of all ceramic crowns and bridges [11–17].

When stress is loaded, ceramics generates very small deformation and results in fracture before plastic deformation. Considering that strength of solid substance is a required stress per unit area up to the point of fracture, area is proportional to required stress up to the point of fracture in metals which are ductile materials; however, such a similarity rule does not come into existence in brittle materials. Therefore, breaking strength evaluation covering brittleness of ceramics is required [18–25], and ISO [26] and JIS [27] standards describe fracture toughness value testing methods for advanced ceramics. However, it has been known that single-edge precracked beam (SEPB) method that is used for these standards is difficult to give experimental cracks, and that it is required to obtain Young's modulus from different experiments. Moreover, different correction factors are used for fracture toughness values obtained for commercially available porcelains by different researchers, and this difference affects test conditions including test load, loading time, and loading rate. Thus, the indentation fracture (IF) method has always been applied as a fracture toughness value testing method in the field of dentistry because test slip

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1883-1958/$ – see front matter
http://dx.doi.org/10.1016/j.jpor.2013.01.005

preparation and the test method are easy and simple. IF method is a method to calculate fracture toughness value based on the lengths of impressions and cracks generated by indentation of a Vickers indenter into mirror polished test specimens. On the other hand, the IF method has characteristics that fracture toughness values cannot be calculated accurately when the load is too heavy because interfacial fractures occur on the test specimens, as well as when the load is too light because insufficient cracks occur [19,23,24]. Therefore, an optimum indentation fracture load should be selected for each different material to be measured.

Zirconia produces about 4% of volume expansion by crystal phase transformation that is caused by transformation of tetragonal crystal stabilized at room temperature to monoclinic crystal by loading. Therefore, development of cracks is prevented because crystal phase transformation from tetragonal crystal to monoclinic crystal accompanied by volume expansion as well as compression stress at the leading end of the cracks are occurred when cracks are grown in zirconia. It is required to evaluate material characteristics of the new material, including how loading changes indentation and crack, and how much the fracture toughness value is improved as a result of the loading in zirconia, which is a high-strength ceramic. It would contribute to clinical application, which has a demand for safety and assurance, if measure of fracture toughness values for such high-strength zirconia would be determined.

In this study, effects of the indentation fracture load on indentation formed by Vickers indenter, crack length, and fracture toughness values were examined using the IF method as a means to evaluate brittleness with the aim of normalization of measuring conditions for zirconia.

2. Materials and methods

2.1. Materials

Zirconia, 3 mol yttrium-stabilized tetragonal zirconia polycrystal (3 mol Y-TZP) (Kavo Everest® Zirconium Soft, Kavo, Biberach, Germany), of composition 5.0 wt% Y_2O_3 to 94.8 wt% ZrO_2, was used as a material of the experiment. 3 mol Y-TZP is usually using the present clinical scene.

2.2. Specimen preparation

Test specimen was prepared at a size of 2 mm (width) × 5 mm (thickness) × 25 mm (length) using a block of semi-sintered body of 20 mm (width) × 20 mm (thickness) × 40 mm (length) (Kavo Everest® Zirconium Soft, Kavo), which was dimensionally corrected with 20.19% of shrinking percentage, by cutting with a low-speed cutter (ISOMET, BUEHLER), followed by calcining with a baking furnace (Kavo Everest® therm) at 1450 °C as a final heating temperature for 10 h according to conditions specified by the manufacturer. End faces of the calcined test specimen were processed in parallel (0.05 mm). Furthermore, the surface of the test specimen was polished with #150, #400, #600, #1200, and #2000 grit waterproof abrasive papers, and then was mirrored with a dedicated buff using 1.0 μm diamond paste.

2.3. Test conditions

Experiment was conducted with the IF method using a Vickers hardness tester (AVK-A, Akashi, Kanagawa, Japan) by pressing a Vickers indenter into the test specimen to generate semicircular or semielliptical, vertical crack around the indentation. Length of this crack was measured, and fracture toughness value (MPam$^{1/2}$) was calculated for the measured value obtained using Niihara's formula [23] as below:

$$K_{IC} = 0.203(c/a)^{-3/2} H a^{1/2}$$

K_{IC}, fracture toughness value (MPam$^{1/2}$); a, 1/2 of indentation diagonal length (μm); c, 1/2 of crack length (μm); and H, Vickers hardness (Hv).

Meanwhile, five conditions of indentation fracture load, 9.8, 49, 98, 196 and 294 N (1, 5, 10, 20 and 30 kgf), were used to examine the effect of difference of loading on fracture toughness value; and load holding time was set to 15 s. Experiments were conducted 10 times for each condition, i.e., total 50 times. Calculated Vickers hardness and fracture toughness values were analyzed with one-way analysis of variance and then multiple comparisons (Scheffe). The significance level was defined as 95%.

2.4. Scanning electron microscope observation

Test specimens after a test using the IF method were observed using a scanning electron microscope (SEM, S-4000, Hitachi, Tokyo, Japan) to examine the appearance of indentation and cracks.

3. Results

3.1. Indentation diagonal lengths, crack lengths, Vickers hardness, and fracture toughness value

Fig. 1 shows indentation diagonal lengths and the effect of indentation fracture load. Average of indentation diagonal lengths (a) occurred by 9.8, 49, 98, 196 and 294 N of indentation fracture load were 15 ± 1.3, 39 ± 0.4, 56 ± 0.4, 81 ± 0.4, and 101 ± 0.7 μm, respectively. The indentation diagonal length was increased with increase of the loading. Correlation between the loading and the indentation resulted in a correlation coefficient of $r = 0.98$ and a regression formula of $y = 2.81x + 21.57$; and was highly significant.

Fig. 2 shows the effect of the indentation fracture load on crack length. Crack lengths (c) at 9.8 and 49 N were not measurable because the cracks were too small to measure with the measuring microscope of the Vickers hardness tester. Average crack lengths (c) at 98, 196 and 294 N were 112 ± 4.4, 192 ± 8.5, and 260 ± 4.6 μm, respectively. The crack length was increased with increase of the loading. Correlation between the loading and the crack length resulted in a correlation