Strength testing variables in dental ceramics
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CHAPTER 4

Fracture toughness determination of two dental porcelains with the indentation strength in bending method

Keywords: Dental porcelain, Fracture toughness, Indentation strength
4.1 Abstract

Objective: This study was to investigate the influence of the bending test configurations and the crosshead displacement speeds on the fracture toughness ($K_{IC}$) of dental porcelains obtained with the indentation strength in bending (ISB) method.

Methods: The strength of the dental veneering porcelains Duceram and Sintagon Zx, Vickers’ indented at a load of 2 kg was measured at a crosshead speed of 0.5 mm/min with three test configurations, which were 3-point, 4-point, and biaxial bending. Two more groups of Sintagon Zx were tested the same way, but at speeds of 0.1, and 0.05 mm/min, respectively. Both porcelains, the three crosshead speeds, and the three test configurations were compared statistically.

Results: Duceram had a higher toughness than Sintagon Zx with all three test configurations and there was no significant difference between three test configurations with either porcelain. Within the crosshead speed groups of Sintagon Zx, a significant difference was found only in the 0.5 mm/min group between the 3-point, and 4-point configurations.

Within the configuration groups, significant differences were found between all speeds with the 3-point configuration and only between the highest and lowest speed with the 4-point and the biaxial tests.

Conclusion: The crosshead displacement speed can cause statistically different results of fracture toughness obtained with the ISB method.
4.2 Introduction

Application of porcelain and ceramic in prosthodontics resulted in a naturally appearing restoration, no matter if it belongs to metal- or all-ceramic restorations, compared with metal restorations [1, 2]. However, dental porcelains and ceramics are brittle materials, which generally fail in tension due to their limited ductility, which restricts the ability to absorb a great deal of elastic strain energy before fracture [3]. A major weakness of these materials is the sensitivity to flaws, which may have developed as a result of thermal, chemical or mechanical processes, and act as local stress raisers. At a certain critical applied stress, a crack can originate from a flaw and propagate, engendering final catastrophic fracture [3-6]. Fracture toughness, $K_{IC}$ is defined as the critical stress intensity level at which a given flaw starts extending and provides insight into the potential resistance to crack growth of a material [7-9]. So in the last decade in the field of dental porcelains and ceramics research, much attention has been paid to the fracture toughness [2].

Fracture toughness is reported to be an intrinsic property of ceramics, which indicates the ability of dental porcelains and ceramics to resist crack extension. So the accurate measurement of $K_{IC}$ is essential. However, determination of $K_{IC}$ is technically rather sensitive, and the obtained values and rankings may be different depending on the techniques and procedures used [8-10].

Among the many methods for fracture toughness determination, the indentation strength in bending (ISB) method as introduced by Chantikul et al. [11], is relatively simple, and reportedly accurate and reproducible, compared to other methods [8, 9, 12, 13]. It uses the bending strength of for example beams, which have an indentation at the center of the tensile surface. There is no need to determine the initial size of the flaw, because an entry of the indentation load in the equation is used instead. Since the 1990s there has been a dramatic increase in the use of this method [14-26]. However, uncertainty may be introduced with the test configuration [27, 28], which influences the result of $K_{IC}$. For example, first, 3-point, 4-point, and biaxial bending tests, lead to different values due to different effective surface areas or volumes subjected to stress. Second, obtained strength values could depend on the crosshead speed.

In this light, the aim of the present study was to compare fracture toughness values obtained for two dental porcelains with the ISB method with different test configurations and crosshead speeds. The assumption subject to investigation was that there was no statistical difference between $K_{IC}$ values obtained with the ISB method, when different test configurations and crosshead speeds were used.
4.3 Materials and Methods

Two veneering dentine porcelains were involved in the comparison: Duceram (Degussa Dental GmbH, Hanau, Germany) and Sintagon Zx (Elephant Dental B.V., Hoorn, The Netherlands) (Table 4.1).

<table>
<thead>
<tr>
<th>Materials</th>
<th>Elastic modulus</th>
<th>Vicker's hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duceram</td>
<td>60.8 (2.4)</td>
<td>4.97 (0.11)</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>64.5 (1.6)</td>
<td>4.43 (0.08)</td>
</tr>
</tbody>
</table>

Table 4.1 Elastic moduli and Vickers’ hardness in GPa and standard deviation in parentheses

Specimen preparation

For the beam specimens in the 3-point and 4-point setups, porcelain blocks were made by condensing the powder a in brass mould (4 mm × 30 mm × 40 mm). The discs for the biaxial tests were formed by condensing the powders in a stainless steel ring with an inner diameter of 21 mm and a height of 2.5 mm.

The blocks and the discs were removed from the mould, and fired in a dental porcelain furnace (STRATOS, Elephant Dental B.V., Hoorn, The Netherlands) in compliance with the manufacturer's recommendations. The cooling time was prolonged to 1 h to accommodate for the relatively large size of the bodies as compared to normal applications. The upper and lower surfaces of the square blocks were ground parallel to a thicknesses of 3 mm and sawed into rectangular beams of 2 mm × 3 mm × 26 mm. The round discs were ground parallel to a thickness of 2 mm and the diameter of approximately 18 mm was left unchanged. All specimens were polished on all surfaces with wet silicon carbide paper of the grits 400, 600, and 1200 successively and the sharp edges were chamfered, except the end planes of the beams and the circumference of the discs, which were left as fired.

The specimens were annealed to release residual stresses by keeping these close to the glass transition temperature for 90 min and cooling to 100 °C for another 90 min. This was 450 °C for Sintagon Zx, and 575 °C for Duceram.

Fracture toughness with the ISB method

Vickers’ indentations were made (HM-124 Hardness Testing Machine, Mitutoyo Corp., Kanagawa, Japan) at the center of the tensile surface of the specimens at a load of 19.6 N during 15 s. The radial cracks, which arise with this load, serve as
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the pre-crack in this test. Within 30 min following the indentation, the specimens were loaded until fracture occurred in a tensilometer (ACTA Intense, ACTA, Amsterdam, The Netherlands) [27-30]. Specimens where the fracture did not originate from the Vickers’ indentation were excluded from the study and testing was continued until at least 10 acceptable test results were acquired.

The fracture toughness was obtained with [11],

\[ K_c = \eta (E / H)^{3/8} (\sigma_f P^{1/3})^{3/4} \]

where \( \eta \) is the geometrical constant (0.59), \( E \) the elastic modulus, \( H \) the Vickers’ hardness, and \( P \) is the indentation load. Calculation of the strength, \( \sigma_f \), depends on the test setup and is discussed later.

For each porcelain the elastic modulus was determined with a 3-point bending test on 20 beams without indentation at a crosshead speed of 0.5 mm/min. The bending deflection of the specimens was recorded. The modulus was calculated with:

\[ E = \frac{WL^3}{4bd^3q} \]

where \( E \) is the elastic modulus, \( W \) the fracture load, \( L \) the span length (20 mm), \( B \) the specimen width, \( D \) the specimen thickness, and \( q \) is the bending deflection.

The Vickers’ hardness (\( H \)) was measured on broken specimens (\( n = 10 \)) using a load of 1.96 N during 15 s, which magnitude prevented the introduction of radial cracks. The hardness was calculated with \( H = 1.854P/(2a)^2 \), where \( P \) is indentation load (1.96 N), and \( 2a \) is the average of the two diagonals of the indentation.

**The test configurations and crosshead speeds**

The three test configurations used, were 3-point, 4-point, and biaxial bending. The last was carried out in a ball-on-ring setup.

The strength with the 3-point, and 4-point bending test was calculated according to the following formula [27]:

\[ \sigma_f = \frac{3WL_1(L_1 - L_2)}{2BD^3} \]
where $\sigma_f$ is the strength, $W$ the fracture load, $L_1$ the supporting span length of 20 mm, $L_2$ the loading span length, which is 10 mm in the 4-point test and 0 mm in the 3-point test, $B$ the specimen width, and $D$ is the specimen thickness.

The strength with the biaxial bending test (ball-on-ring) was calculated as the following equation:

$$\sigma_f = \frac{3W(1 + \nu)[1 + 2 \ln(D_s / b) + \{1 - (b^2 / 2D_s^2)\}((D_s / D)^2 - 1 - \nu))/2]}{4\pi B^2}$$

where $\sigma_f$ is the strength, $W$ the fracture load, $D_s$ the diameter of the support circle (16 mm), $b$ the diameter of the area with uniform load ($=2B/3$), $B$ the thickness, and $D$ is the diameter of disc. For $\nu$, the Poisson's ratio, a value of 0.25 was used, as recommended with ISO 6872 [28-30].

Sintagon Zx was tested in all three configuration with crosshead speeds of 0.05, 0.1, and 0.5 mm/min. Duceram was tested only at 0.5 mm/min.

**Statistic analysis**

The fracture toughness values among different groups were subject to two-way ANOVA and pair wise comparison, where a $p$-value of less than 0.05 was considered statistically significant. The comparisons were carried out between all groups of Sintagon Zx, between the configurations with Duceram, and between Duceram and Sintagon Zx at 0.5 mm/min.

### 4.4 Results

Elastic moduli and Vickers’ hardness values are listed in Table 4.1 and the fracture toughness values and standard deviations are listed in Table 4.2.

**Table 4.2** ISB fracture toughness in MPa m$^{-1/2}$ with standard deviation in parentheses

<table>
<thead>
<tr>
<th>Bending test methods</th>
<th>Crosshead speed (mm/min)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.05</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>Duceram</td>
<td>Sintagon Zx</td>
</tr>
<tr>
<td>3-Point</td>
<td>1.100 (0.068)</td>
<td>0.885 (0.064)</td>
</tr>
<tr>
<td>4-Point</td>
<td>1.099 (0.065)</td>
<td>0.829 (0.041)</td>
</tr>
<tr>
<td>Biaxial</td>
<td>1.095 (0.083)</td>
<td>0.850 (0.047)</td>
</tr>
</tbody>
</table>
With all three test configurations the ISB $K_{IC}$ values for Duceram were significantly higher than for Sintagon Zx and no significant difference existed between the test configurations with either porcelain in Table 4.3 and Table 4.4.

**Table 4.3**  Pairwise comparison of the configuration effect within the material effect with Duceram and Sintagon Zx

<table>
<thead>
<tr>
<th>Material</th>
<th>Configurations</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duceram</td>
<td>3-Point vs. 4-point</td>
<td>0.975</td>
</tr>
<tr>
<td></td>
<td>3-Point vs. biaxial</td>
<td>0.848</td>
</tr>
<tr>
<td></td>
<td>4-Point vs. biaxial</td>
<td>0.872</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>3-Point vs. 4-point</td>
<td>0.051</td>
</tr>
<tr>
<td></td>
<td>3-Point vs. biaxial</td>
<td>0.215</td>
</tr>
<tr>
<td></td>
<td>4-Point vs. biaxial</td>
<td>0.462</td>
</tr>
</tbody>
</table>

The differences are not statistically significant at the 0.05 level.

**Table 4.4**  Pairwise comparison of the material effect within the configuration effect between Duceram and Sintagon Zx

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Materials</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Point</td>
<td>Duceram vs. Sintagon Zx</td>
<td>$&lt;0.001^*$</td>
</tr>
<tr>
<td>4-Point</td>
<td>Duceram vs. Sintagon Zx</td>
<td>$&lt;0.001^*$</td>
</tr>
<tr>
<td>Biaxial</td>
<td>Duceram vs. Sintagon Zx</td>
<td>$&lt;0.001^*$</td>
</tr>
</tbody>
</table>

$^*$ The difference is statistically significant at the 0.05 level.

No significant difference was found between the test configurations within the speed groups in Table 4.5, except at 0.5 mm/min between the 3-point and 4-point tests.

The 3-point test showed significant differences between all crosshead speeds in Table 4.6. Both the 4-point and the biaxial configuration produced a significant difference only between the 0.05 and the 0.5 mm/min group.
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Table 4.5  Pairwise comparison of the configuration effect within the crosshead speed effect of Sintagon Zx

<table>
<thead>
<tr>
<th>Crosshead speed</th>
<th>Configurations</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05 mm/min</td>
<td>3-Point vs. 4-point</td>
<td>0.958</td>
</tr>
<tr>
<td></td>
<td>3-Point vs. biaxial</td>
<td>0.263</td>
</tr>
<tr>
<td></td>
<td>4-Point vs. biaxial</td>
<td>0.297</td>
</tr>
<tr>
<td>0.1 mm/min</td>
<td>3-Point vs. 4-point</td>
<td>0.094</td>
</tr>
<tr>
<td></td>
<td>3-Point vs. biaxial</td>
<td>0.656</td>
</tr>
<tr>
<td></td>
<td>4-Point vs. biaxial</td>
<td>0.227</td>
</tr>
<tr>
<td>0.5 mm/min</td>
<td>3-Point vs. 4-point</td>
<td>0.026*</td>
</tr>
<tr>
<td></td>
<td>3-Point vs. biaxial</td>
<td>0.158</td>
</tr>
<tr>
<td></td>
<td>4-Point vs. biaxial</td>
<td>0.403</td>
</tr>
</tbody>
</table>

* The difference is statistically significant at the 0.05 level.

Table 4.6  Pairwise comparison of the crosshead speed effect within the configuration effect of Sintagon Zx

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Crosshead speeds</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-Point</td>
<td>0.05 vs. 0.1 mm/min</td>
<td>0.007*</td>
</tr>
<tr>
<td></td>
<td>0.05 vs. 0.5 mm/min</td>
<td>0.000*</td>
</tr>
<tr>
<td></td>
<td>0.1 vs. 0.5 mm/min</td>
<td>0.017*</td>
</tr>
<tr>
<td>4-Point</td>
<td>0.05 vs. 0.1 mm/min</td>
<td>0.335</td>
</tr>
<tr>
<td></td>
<td>0.05 vs. 0.5 mm/min</td>
<td>0.008*</td>
</tr>
<tr>
<td></td>
<td>0.1 vs. 0.5 mm/min</td>
<td>0.079</td>
</tr>
<tr>
<td>Biaxial</td>
<td>0.05 vs. 0.1 mm/min</td>
<td>0.276</td>
</tr>
<tr>
<td></td>
<td>0.05 vs. 0.5 mm/min</td>
<td>0.014*</td>
</tr>
<tr>
<td></td>
<td>0.1 vs. 0.5 mm/min</td>
<td>0.165</td>
</tr>
</tbody>
</table>

* The difference is statistically significant at the 0.05 level.
4.5 Discussion

Although in some literature the ISB method has shown good agreement with conventional fracture mechanics tests [8, 9, 11-13], the consistency of the results with standard methods were mixed [8-10, 14, 20]. The fracture toughness, determined with the ISB method, showed dependence on the indentation load, in which a higher indentation load leads to an apparently higher $K_{ic}$ value [8, 16, 17]. This dependence on indentation load provides information on the $R$-curve behavior of dental ceramics [31].

Test configuration

In dental literature, few investigations evaluated the influence of the configuration of the test, as 3-point, 4-point and biaxial bending tests could be used in the ISB test. These test configurations may demonstrate different strength values in general because different areas and volumes are exposed to stress [27, 28]. Although the indentation introduced flaw, which fixates the point of cracking, should theoretically prevent this influence, the role of the tests configuration in the ISB results is uncertain. Furthermore, alignment errors of the load point with the indentation contribute to scatter in the data with the 3-point and the biaxial test.

Albakry et al. [20] have found a significant difference in the ISB fracture toughness between the 3-point and the biaxial tests with Empress 2 and their experimental glass–ceramic, two all-ceramic substrate materials with much higher $K_{ic}$ values. Nevertheless, Empress with a low toughness of about 1.3 MPa m$^{1/2}$ had very close ISB $K_{ic}$ values in those two test configurations. In the present study, with a crosshead speed of 0.5 mm/min, all test configurations, that is 3-point, 4-point, and biaxial bending displayed consistent ISB fracture toughness values for both Duceram and Sintagon Zx, which materials had low toughness values too. This seems to suggest that with low fracture toughness porcelains and ceramics the ISB results with different test configurations are comparable, while significant differences between the configurations may occur with ceramics of higher toughness. If this is true, further investigation should be given to the role of the test configurations in ISB fracture toughness determination, since dental porcelains and ceramics have a large range of fracture toughness and a distinct diversity in chemistry and microstructure [2, 34]. More different types and varieties of dental porcelains and ceramics should be investigated in ISB $K_{ic}$ measurement with different strength tests and their comparability to standard methods.
Crosshead speed

In bending procedures the strength is known to increase with crosshead speed, which is the basis for the determination of slow crack growth parameters with a dynamic test [32, 33].

In the present study it is understandable that at a lower loading speed, more time is available for the stored strain energy to produce crack growth, leading to slower crack propagation with lower stress. On the contrary, at a higher load speed, a material needs more energy to drive the growing crack [27, 32].

In dental literature, mostly crosshead speeds of 0.5 and 0.1 mm/min have been used. In the current investigation, crosshead speeds ranging from 0.5 to 0.05 mm/min were used, which covered the range of speeds often used in ISB test and it was found that a high load speed elevated the ISB fracture toughness. In other words, a high loading speed causes overestimation of $K_{ic}$ while it may be underestimated with a low crosshead speed. Despite the absence of many significant differences, all test configurations, especially 3-point bending test manifested obvious sensitivity to crosshead speed. Based on the present study, such a crosshead speed range may cause confusion in comparison even of values within the ISB method itself.

4.6 Conclusion

More different types of dental porcelains and ceramics with a diversity in fracture toughness, chemistry and microstructure should be involved in ISB fracture toughness determination and comparison in order to investigate the comparability of the ISB method to recognized standards when different test configurations are used.

Fracture toughness results, obtained with the ISB method are sensitive to the crosshead speed, which may lead to statistically different and perhaps confusing results.

4.7 References

Fracture toughness determination of two dental porcelains


