Strength testing variables in dental ceramics

Wang, H.

Citation for published version (APA):
CHAPTER 5

Indentation-strength fracture toughness: the role of the indentation load at smaller flaw sizes

Keywords: Dental porcelains, Fracture toughness, Indentation strength, flaw size
5.1 Abstract

Objective: The aim of the study was to investigate the influence of small indentation loads on ISB fracture toughness determination of dental veer ceramics.

Materials and Methods: Duceram Plus and Sintagon Zx were tested in 3-point, 4-point, and biaxial bending while a soda lime glass was tested in 4-point bending as a reference. Duceram Plus was indented at 9.8, and 19.6 N, Sintagon Zx at 4.9, 9.8, and 19.6 N, and glass at 1.96, 2.94, 4.9, 9.8, and 19.6 N. Unindented specimens were tested, too. The specimens which fractured at a flaw other than the precrack were rejected.

Results: All toughness values increased significantly with indentation load. Duceram Plus was significantly tougher than Sintagon Zx and glass. No statistical differences were found between the setups, except with Duceram Plus in the 4-point setup, which produced lower toughness values. At a fixed indentation load, different materials are compared at different flaw sizes, where tougher materials have smaller indentation flaws and more rejections. At small flaw sizes, rejected ISB specimens are often stronger than average.

Conclusions: Testing the unindented strength of identical specimens to verify that the indented strength is sufficiently lower is a safer requirement for a valid experiment than (only) setting a maximum to the number of rejected specimens.
5.2 Introduction

Fracture phenomena such as chipping, delamination, or complete failure, are a major reason for failure of all-ceramic dental restorations [1-5]. Cores of strong ceramics like zirconia increasingly replace metal cores underneath veneer porcelains and new phenomena occur. For example, metal alloy core materials are perhaps half as strong as good quality zirconia but depending on restoration design, type of loading, and other circumstances, cracks may initiate in the much weaker veneer, propagate into the ceramic core and cause complete fracture [4, 5]. Perhaps not surprisingly, a good quality, well-fused bond between the core and the veneer increases this problem. The strength is a rather variable property of these brittle materials, which is determined by two other properties; the presence of irregularities, flaws, roughness, scratches, etc. that lead to stress concentrations and two, the fracture toughness or $K_{Ic}$, which is a material constant. Flaws may be related to a composite structure and as such be a material constant, but surface roughness and scratches generally are related to the finishing procedure or service life [6, 7].

The toughness, i.e. the resistance to crack extension is not easily assessable and many techniques have been developed to measure the fracture toughness of brittle materials. A few methods, such as the Single-Edge-Pre-cracked-Beam (SEPB), the Chevron-Notched beam or rod (CN), or the Surface-crack-by-Flexure (SCF) produce consistent and reproducible $K_{Ic}$ values and have been accepted by standardization organizations such as the ASTM or the ISO [8-11]. Many other methods, the Indentation-strength in Bending (ISB), Indentation Fracture (IF), etc, are more convenient or easier to perform, but lead to a variety of toughness values depending on many factors [10-24]. The toughness of dental porcelains and ceramics has been evaluated with various test methods [14-25].

The Indentation-Strength-in-Bending (ISB) method, which is often used because of its convenience, seems to be an appropriate test to evaluate the fracture toughness of dental ceramics. However, differences in micro-structural properties of the tested materials [17, 18, 23], the crosshead speed [24], as well as the indentation load [15, 16] have been shown to clutter the resulting ISB-toughness in a complex way. With this method the tensile surfaces are polished and precracked with a Vickers’ indenter. The radial cracks at the indentation are considered a reasonable reproduction of typical surface damage [12]. After the tests, the fracture surfaces are examined to verify that failure occurred from the indentation and the toughness is obtained with a formula, which uses the failure stress, the precrack force, the Vickers’ hardness, and the elastic modulus. The hardness may be measured on broken specimens the elastic
modulus requires extra experiments with unindented specimens. A minimum precrack load generally is required to prevent a large amount of rejected specimens because a smaller precrack increases the chance that another, already present flaw is effectively larger and that fracture occurs there and not at the precrack.

This aim of this study was to investigate the influence of small indentation loads on ISB experiments with relatively weak dental veneer ceramics in the 3-point, 4-point, and biaxial bending tests, respectively.

5.3 Materials and Methods

Apart from a soda lime glass sheet material, which was used as a reference, the dentin powders of two veneer ceramic systems were tested. These silicates or glass ceramics are intended to increase the strength of the final structure and provide opacity to mask the core. Leucite particles with a high thermal contraction coefficient are added to match the thermal contraction of the core material and to increase the toughness. Duceram Plus (Degussa Dental GmbH, Hanau, Germany, LOT: 41598) is used with porcelain-fused-to-metal crowns, Sintagon Zx (Elephant Dental B.V., Hoorn, The Netherlands, LOT: A-1811) is intended for zirconia all-ceramic systems.

Specimen preparation

The powders with their mixing liquids were condensed in brass moulds of 30 x 50 x 4 mm³ for the 3- and 4-point tests and of 24 mm diameter x 2.5 mm thickness for the ball-on-ring tests. The blocks were removed from the moulds and fired in a dental oven (STRATOS, Elephant Dental B.V., Hoorn, The Netherlands) according to the manufacturers’ instructions. The cooling time was extended to 30 min to avoid thermal stresses. The upper and lower surfaces of the fired blocks were ground parallel with 30/80 diamond paste in a grinding device (VEM Metallurgy, Vos & Van Eijk Metallurgie B.V., Houten, the Netherlands). The rectangular blocks were reduced to a thickness of 3.0 mm and the round discs to 1.9 mm.

The blocks were cut into bars (2.0 height x 3.0 width x 25.0 mm³) with a 0.5 mm diamond saw (ISOMET Q2 1000, Buehler Ltd, Lake Bluff, IL). The glass beams (2.0 height x 3.9 width x 25 mm³) were cut from 3.9 mm sheet material. The tensile surface of all bars and discs were polished with wet silicon carbide papers of 400, 600, and 1200 grit successively, and the sharp edges of the rectangular bars were chamfered. The specimens of glass and Sintagon Zx were annealed at 450°C and the Duceram Plus specimens at 600°C during 90 min and cooled to 100°C in another 90 min. The roughness, i.e. the Rₐ value was measured in three places on six random
beams of two series of each material (SZ 700 profilometer, Mitutoyo Corp., Kanagawa, Japan).

**Strength testing**

Vickers’ indentations were made (HM-124 Hardness Testing Machine, Mitutoyo Corp., Kanagawa, Japan) in the middle of the tensile surfaces. Unindentected specimens of each material were tested as well. Duceram Plus specimens were indented at 9.8 and 19.6 N, Sintagon Zx specimens at 4.9, 9.8, and 19.6 N, and the glass beams at loads of 1.96, 2.94, 4.9, 9.8, and 19.6 N. The radial cracks underneath the Vickers’ indentation serve as the pre-crack in this test. Because of the residual stresses around the indentation the pre-cracks continue to grow during the first few minutes following the indentation. The specimens were loaded after 20 to 30 min at 0.05mm/min until fracture occurred in a tensilometer (ACTA Intense, ACTA, Amsterdam, NL). Specimens, where the fracture did not originate at the Vickers’ indentation were excluded from the toughness but not from the strength assessments and testing was continued until at least 10 acceptable toughness results were obtained.

**Setup configurations**

The two dental veneers were tested in three configurations: 3- and 4-point bending of bars, and biaxial bending of discs in a ball-on-ring setup. The glass beams were tested in 4-point bending. In the 3- and 4-point bending tests the strength was calculated according to [26]:

\[
\sigma_f = \frac{3W(L_1 - L_2)}{2BD^2}
\]

Where \(\sigma_f\) is the strength, \(W\) is the fracture load, \(L_1\) the support span (20.0 mm), \(L_2\) the loading span (10.0 mm for 4-point bending, 0 mm for 3-point bending), \(B\) the specimen width, and \(D\) the specimen thickness.

Strength with the ball-on-ring test was calculated with the following equation:

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

Where \(\nu\) is the Poisson’s ratio, \(D_s\) the diameter of the support ring (16.0 mm), \(D\) the diameter of disc (21 mm), \(T\) the thickness, and \(b = 2T/3\) is the diameter of the area with
a uniform load. A value of 0.25 was used for Poisson’s ratio, as recommended by ISO 6872 [27, 28]. The diameter of the loading ball is 5 mm. The support ring is a circle of 16 cylindrical 2.5 mm pins with rounded tops. The pins are supported as pistons entering a shared and sealed compartment filled with soft (Shore 10) silicone rubber, which works as a non-leaking hydraulic fluid. Discs, which are not absolutely flat, for example when surfaces are left as fired, may also be tested this way.

The elastic modulus was determined with the 3-point bending setup on ten bars without indentation but at a crosshead speed of 0.5 mm/min.

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

Where \( E \) is the elastic modulus, \( L \) the support span (20.0 mm), and \( q \) the bending deflection.

The Vickers’ hardness, \( H_v \), was measured on broken specimens (\( n = 10 \)) using an indentation load, \( P \), of 1.96 N, with \( H = 1.854P/l^2 \), where \( l \) is the average of the two diagonals of the indentation. The ISB fracture toughness was obtained with the ISB equation [12]:

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

Where \( \eta \) is the geometrical constant (0.596), \( E \) the elastic modulus, \( H \) the Vickers’ hardness, and \( P \) the precrack load. The geometrical constant is slightly greater than the 0.59 used by Chantikul et al., because they used 2 instead of 1.854 in the Vickers’ equation.

Three-way analysis of variance (ANOVA) and SNK pair wise comparison was carried out to assess the individual and interaction effect of materials, strength test configurations, and indentation loads on the fracture toughness, at a significance level of 0.05. The software used was SPSS 10.0 (SPSS inc., Chicago, USA).

5.4 Results

Figure 5.1 shows the toughness and Figure 5.2 the strength values, which are listed numerically in Table 5.1 The table also lists the numbers of bars and strength of the bars, which were rejected for the calculation of the ISB toughness. Note that the average values in the stress column do include the values of the rejected bars. In all cases the reason for rejection was fracture at another flaw than the indentation.
Table 5.1  Average ISB toughness and strength values, numbers of specimens tested and rejected and strength of each rejected specimen. The standard deviations are in parentheses. The elastic modules, E, and Vickers’ hardness’s, HV, are in GPa. Not significant differences as connected with index letters with 0N indentation load series are based on strength values, all others on toughness values.

<table>
<thead>
<tr>
<th>Configuration</th>
<th>ISB values</th>
<th>Specimens tested and rejected</th>
<th>Strength of rejected bars – MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Material</td>
<td></td>
<td>N</td>
</tr>
<tr>
<td>0</td>
<td>4-p Duceram Plus</td>
<td>9.8</td>
<td>0.825(0.032)$^{a}$</td>
</tr>
<tr>
<td>19.6</td>
<td>0.923(0.067)$^{b}$</td>
<td>44.9(4.3)</td>
<td>14/3</td>
</tr>
<tr>
<td>0</td>
<td>3-p E=61.6(1.4)</td>
<td>9.8</td>
<td>0.891(0.037)</td>
</tr>
<tr>
<td>19.6</td>
<td>-</td>
<td>50.2(4.4)</td>
<td>11/0</td>
</tr>
<tr>
<td>0</td>
<td>0.909(0.061)$^{c}$</td>
<td>55.4(5.0)</td>
<td>15/4</td>
</tr>
<tr>
<td>19.6</td>
<td>1.01(0.047)</td>
<td>50.5(3.1)</td>
<td>12/0</td>
</tr>
<tr>
<td>0</td>
<td>4-p Sintagon Zx</td>
<td>9.8</td>
<td>0.667(0.073)</td>
</tr>
<tr>
<td>19.6</td>
<td>0.717(0.060)</td>
<td>38.9(4.4)</td>
<td>11/1</td>
</tr>
<tr>
<td>0</td>
<td>-</td>
<td>33.2(4.8)</td>
<td>11/1</td>
</tr>
<tr>
<td>4.9</td>
<td>0.761(0.047)</td>
<td>33.2(4.8)</td>
<td>11/1</td>
</tr>
<tr>
<td>9.8</td>
<td>19.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>3-p E=64.5(1.6)</td>
<td>4.9</td>
<td>0.675(0.044)</td>
</tr>
<tr>
<td>9.8</td>
<td>0.725(0.039)$^{d}$</td>
<td>39.4(2.7)</td>
<td>12/2</td>
</tr>
<tr>
<td>19.6</td>
<td>0.760(0.069)$^{d}$</td>
<td>33.3(4.0)</td>
<td>11/0</td>
</tr>
<tr>
<td>0</td>
<td>-</td>
<td>46.7(3.1)</td>
<td>8</td>
</tr>
<tr>
<td>4.9</td>
<td>0.652(0.060)</td>
<td>43.1(5.2)</td>
<td>11/1</td>
</tr>
<tr>
<td>9.8</td>
<td>0.716(0.025)</td>
<td>38.7(1.8)</td>
<td>10/0</td>
</tr>
<tr>
<td>19.6</td>
<td>0.787(0.042)</td>
<td>34.9(2.5)</td>
<td>10/0</td>
</tr>
<tr>
<td>0</td>
<td>-</td>
<td>96.4(11.6)</td>
<td>8</td>
</tr>
<tr>
<td>4-p Glass</td>
<td>E=58.6(3.5)</td>
<td>1.96</td>
<td>0.645(0.044)$^{e}$</td>
</tr>
<tr>
<td>2.94</td>
<td>0.652(0.024)$^{e}$</td>
<td>57.2(10.2)</td>
<td>12/2</td>
</tr>
<tr>
<td>4.9</td>
<td>0.674(0.019)$^{ef}$</td>
<td>47.2(1.7)</td>
<td>12/0</td>
</tr>
<tr>
<td>9.8</td>
<td>0.708(0.027)$^{f}$</td>
<td>40.0(2.0)</td>
<td>12/0</td>
</tr>
<tr>
<td>19.6</td>
<td>0.778(0.042)</td>
<td>36.0(2.6)</td>
<td>12/0</td>
</tr>
</tbody>
</table>
Figure 5.1  ISB toughness values of all series

Figure 5.2  ISB indented strength of all series
The average $R_a$ values of all materials are similar, 0.05 to 0.06 $\mu$m. The toughness values of Duceram Plus at 9.8 N precrack load in the 4-point and in the biaxial configuration are not acceptable, because of the large number of rejected specimens.

All ISB-$K_{IC}$ values increase with indentation load. In all cases the differences between the lowest (≠0) and highest precrack loads are significant, but the differences between the successive steps of indentation load often are not. In all test configurations and indentation loads the ISB toughness values of Duceram Plus are statistically higher than those of Sintagon Zx and the glass. The differences between the toughness values of glass and Sintagon Zx are not significant. The differences between the setup configurations are not significant, except the 4-point setup with Duceram Plus, which produced lower $K_{IC}$ values with both indentation loads. The failure stresses of all indented specimens were significantly lower than the unindented ones with the exceptions of Duceram Plus in the 4-point setup and at 9.8 N in the biaxial setup, where the differences were not significant. A total of 26 rejected specimens are listed. 20 of these, or about three out of four, are stronger than the average of the series in the stress column. On average of all 26, the failure stresses of the rejected specimens are about 14% greater than the average of each one’s series.

The results of the unindented specimens are rather different. Glass is considerably stronger than the dentin veneers, between which the difference varies with the setup, although Sintagon Zx is significantly stronger than Duceram Plus in the 4-point setup. All differences between the setup configurations are significant, except between the 3-point and the biaxial tests with Duceram Plus and between the three bending tests with Sintagon Zx.

### 5.5 Discussion

The results confirm [15, 16] a structural positive influence of the indentation load on the ISB toughness values of the tested materials, which is not caused by a rising R-curve [29]. The increases as shown in Figure 5.1, must be attributed to the ISB method, because especially glass has a flat R-curve. The influence of the precrack load on the apparent toughness seems reasonably linear.

In previous, Chevron Notch (CN) experiments [14, 25] the toughness values of glass and Sintagon Zx were the same as well. The value of 0.70MPa·m$^{1/2}$ in those CN experiments suggests that these materials should be tested at a precrack load just less than 9.8 N in order to obtain correct ISB values (Figure 5.1). The Sintagon Zx was of
the same batch number as the material in the present experiments, the glass came with the same delivery and should be identical too.

**Materials**

The unindented strengths of the veneers are so much less than that of the glass, because their composite structure gives rise to stress concentrations similar to those at other flaws. The leucite particles, which are added for this purpose, have a much greater thermal contraction coefficient than the glassy matrix. This generates residual thermal stresses after annealing and sometimes cracks in the matrix around the particles [30], which work as a minimum flaw size and are known to decrease the strength at smaller flaw sizes including the unindented strength but also to increase the toughness [31]. Moreover some porosity is inevitable with the powder-liquid technique [32,33]. The pores found in Duceram Plus were larger and more numerous than in Sintagon Zx. Sintagon Zx seems to contain just enough leucite to reduce the unindented strength to a level similar to that of Duceram Plus, but not enough to make it any tougher than glass.

Although under different conditions the veneers may be more difficult to polish than glass also because of their structure, grinding the specimens with wet silicon carbide paper up to grit 1200 seems to produce a fairly constant roughness with an average $R_a$ value of 0.05 to 0.06 μm for all materials tested. The difference in unindented strength between glass and the veneers therefore cannot be attributed to the roughness, but largely is related to irregularities, such as pores or leucite particles, present near or cut at the polished tensile surfaces.

**Precrack size**

The strength plot (Figure 5.2) repeats that strength is not a physical constant of these materials. This figure also gives some idea of the damage tolerance of the materials, although at the same precrack load the resulting flaw size is actually smaller with the tougher material. At a fixed indentation load different toughness materials are ranked at different flaw sizes, which could mean that extra test are necessary to establish an appropriate precrack load for each material when the flaw sizes should be similar. Single materials on the other hand may be tested at a range of precrack loads or flaw sizes. The results of Duceram Plus and glass confirm that more rejections occur at smaller flaw sizes.

A fixed precrack load amplifies variations in the toughness of the specimens, as a tougher specimen is stronger, not only because it is tougher but also because the flaw
is smaller. This increases the differences between materials as well as the data scatter within the series, but these phenomena are compensated when the strength values are converted to toughness values with the ISB equation. At smaller sizes of the precrack, this mechanism increases the chance that rejected specimens are stronger than the average of the series, because the smaller precracks in tougher specimens increase the chance of failure at another flaw. Moreover the stress required for fracture at the precrack should be greater than the failure stress of these specimens. Rejecting these specimens of the toughness assessment therefore should decrease the resulting average more than leaving these in.

**Unindented strength**

The results of Duceram Plus at 9.8N, with a large amount of rejected specimens in all setups, illustrate that before or without the indentation the specimens should be sufficiently stronger (**Figure 5.2**) than is required to indentedly fracture at the strength for the appropriate toughness value, as the numbers of rejected specimens, 2, 4, and 6, seems to agree with the ratios of the indented strength to the unindented strength, 89%, 98%, and 104%, in the 3-point, biaxial, and 4-point setup respectively in Table 1. This looks consistent with the much smaller number of rejections with Sintagon Zx at 4.9N where the strength of the indented specimens is 72% (3-point), 84% (4-point), and 92% (biaxial) of the strength of the unindented specimens.

In **Figure 5.2** the strength values of Duceram Plus in the 4-point setup show that indentations simply do not increase the strength of the specimens, which prevents the 4-point setup to produce toughness values in **Figure 5.1** similar to those of the 3-point and biaxial tests. The Duceram Plus 19.6 N 4-point toughness value would just be acceptable, considering the three rejected specimens, but the comparison with the unindented strength in **Figure 5.2** shows that it clearly isn’t acceptable, because the precrack load is much to low. It is not clear however why a majority of the specimens still fractured acceptably at the indentation.

Apparently, the precrack load should be high enough to decrease the strength to a value, which is less than 80 to 90% of the unindented strength, for an acceptable test and not only to prevent rejections due to fracture at another flaw [12].

**Setup configurations**

The number of bars that have to be rejected because fracture occurs at another flaw than the precrack is greater in the 4-point setup because a greater part of the bars is loaded at the maximum stress than in the 3-point setup.
For the same reason, the strength of the unindented specimens is greater in the 3-point setup. It is not very clear how much area is effectively loaded to the maximum stress when the 3-point and the biaxial setup are compared [34, 35]. In the biaxial tests a 1.3 mm circle with a diameter of two thirds of the 1.9 mm thickness of the disc is loaded at the maximum stress. In the 3-point setup this is only a very thin 3mm line opposing the middle roller, but for example 1 mm away from this line the stress is still 90%. Moreover, materials, flaws, and indentations do not necessarily respond similarly to uni- or biaxial stresses. At biaxial stress the crack tip is exposed to perpendicular stress too and with the Vickers’ indentation both radial cracks are loaded, which yields an extra chance on fracture. Fracture from internal flaws, which may occur with unindented specimens should also be sensitive to the rate at which the stress decays towards the neutral line, which then becomes equivalent to the volume of the subsurface part loaded at the maximum stress, which is proportional to the thickness. With both veneers the unindented strength is greater in the 3-point tests. With Duceram Plus the unindented strength in the biaxial setup is not much less than in the 3-point setup, but with Sintagon Zx it is even less than in the 4-point test.

5.6 Conclusion

The present study supports the following conclusions.

- ISB toughness values of glass ceramics increase with the used indentation load.
- At a fixed indentation load, different materials are compared at different flaw sizes.
- At small indentation loads, rejected ISB specimens, which fracture at another flaw than the precrack, often are stronger than specimens, which fracture acceptably.
- Accepting rather than rejecting one or two such specimens might be more accurate.
- Testing the unindented strength of identical specimens to verify that the indented strength is sufficiently lower is a safer requirement for a valid experiment than setting a maximum to the number of rejected specimens.

5.7 References


