Strength testing variables in dental ceramics
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CHAPTER 3

Fracture toughness comparison of three test methods with four dental porcelains

Keywords: Dental porcelains, Fracture toughness, Chevron notched, Indentation strength, Single-edge-notched beam
3.1 Abstract

Objective: The aim of this study was to compare three fracture toughness test methods, using four commercial dental porcelains.

Materials and methods: The fracture toughness test techniques involved were: the single-edge-notched beam (SENB), the indentation strength method (IS), and a rather convenient ASTM standard for advanced ceramics, which is still rarely used in dental ceramic research, the Chevron-notched beam method (CN). Duceram, Duceram LFC, Sintagon Zx and Carrara Vincent were chosen for study. Data was analyzed by two-way and paired ANOVA.

Result: No statistical difference was found between the CN and SENB methods with four dental porcelains, but IS was not always in statistical agreement with SENB or CN. Statistical agreement among all three methods occurred only with Duceram LFC.

Conclusion: The different test methods did not always lead to the same ranking or values of fracture toughness. Yet the toughness results of the SENB method were comparable to those of the CN method for all the four dental porcelains tested in this study.
3.2 Introduction

Ceramics have been increasingly employed in dental restorations because of their esthetics and biocompatibility. Because these materials are very brittle they are also vulnerable to tensile stress, which opens small pre-existing defects, which work as stress raisers. Smoothly finished ceramics are often very strong, but scratches and other small defects, which cumulate with time, seriously degrade the strength of these materials. With little simplifications, a crack initiates or grows when the stress intensity \( K_I \) around a defect or crack tip exceeds the fracture toughness \( K_{IC} \), which is a material property [1-4]. The magnitude of the stress intensity depends on the size and shape (tip radius) of the pre-existing defect or crack, and the applied stress.

In dentistry, increasing attention has been paid to \( K_{IC} \). The research has mainly been focused on two issues: studies into the fracture toughness test methods, and to use the methods to rank dental materials. As many test methods of fracture toughness have limitations, it is essential to assess the consistency of the various techniques by comparing the values obtained with various dental ceramics [3, 4].

A rather convenient ASTM standard for advanced ceramics, which is still rarely used in dental research, is the Chevron-notched beam method (CN). It was hypothesized that this method might be a better method for determining the \( K_{IC} \) of dental ceramics than the more popular single-edge-notched beam (SENB) or the indentation strength method (IS). The aim of this study is to assess the comparability of these three test techniques with four dental veneering porcelains.

3.3 Materials and Methods

The dentin powders of four veneer ceramic systems were used. These were silicates or glass ceramics, reinforced with leucite crystals. Dentin porcelains are intended to add color and should provide opacity to mask the core material. Duceram and Duceram LFC (Degussa Dental GmbH, Hanau, Germany) are used with metal–ceramic systems. Sintagon Zx (Elephant Dental B.V., Hoorn, The Netherlands) is bonded to zirconia all-ceramic substrates. Carrara Vincent (Elephant Dental B.V., Hoorn, The Netherlands) has been designed for metal–ceramic and for Carrara Pressable Core (CPC) all-ceramic systems.

Three fracture toughness test techniques were compared, the indentation strength method (IS), the Chevron-notched beam method (CN) and the single-edge-notched beam method (SENB). Beam-shaped specimens were prepared for at least 10 measurements for each test method. The test configuration is listed in Table 3.1.
Table 3.1 The test configuration of three measurement techniques.

<table>
<thead>
<tr>
<th>Ke test method</th>
<th>CN</th>
<th>SENB</th>
<th>IS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Precrack processing</td>
<td>sawed notch</td>
<td>sawed notch</td>
<td>Vickers’ indentation radial cracks from indentation</td>
</tr>
<tr>
<td>Precrack type</td>
<td>growing crack</td>
<td>large crack (notch)</td>
<td></td>
</tr>
<tr>
<td>Fracture plane</td>
<td>Chevron triangle</td>
<td>half section rectangle</td>
<td>whole section rectangle</td>
</tr>
<tr>
<td>Proportions (B:W:S)</td>
<td>2:3:12</td>
<td>1:2:8</td>
<td>2:3:22</td>
</tr>
<tr>
<td>Dimensions (BxWxL)</td>
<td>3×4.5×26 mm</td>
<td>3×6×26 mm</td>
<td>2×3×25 mm</td>
</tr>
</tbody>
</table>

Specimen preparation

The porcelain powders were condensed in a brass mold (4 mm × 30 mm × 40 mm). The blocks were removed from the mold, 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 the relatively large size of the blocks as compared to normal applications. The upper and lower surfaces of the fired porcelain blocks were ground parallel and the blocks were sawn into rectangular beams (Table 3.1). The specimens were polished with wet silicon carbide papers (grits 400, 600, and 1200, successively) on all surfaces except the end planes, and the sharp edges were chamfered. 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 Duceram LFC, Carrara Vincent, and Sintagon Zx, and 575 °C for Duceram.

Indentation strength method (IS)

Vickers indentations were made (HM-124 Hardness Testing Machine, Mitutoyo Corp., Kanagawa, Japan) in the middle of the tensile surfaces of the beams at a load of 19.6 N. The radial cracks, which arise from this load, serve as the pre-cracks in this test. Because these cracks continue to grow during the first few minutes following indentation, the beams were loaded after 20–30 min in a three-point bending set up at 0.05 mm/min until fracture occurred in a tensilometer (ACTA Intense, ACTA, Amsterdam, The Netherlands). 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 obtained. The fracture strength ($\sigma_f$) of indented specimens was calculated according to following formula:

$$\sigma_f = \frac{3FS}{2WB^2}$$

where $W$ is the fracture load; $L$ the span length; $b$ the specimen width; $d$ is the specimen thickness.

For each material, the elastic modulus ($E$) was determined with a three-point bending test on beams without indentation ($n = 10$). The bending deflection ($q$) of the specimens loaded until failure was recorded. The modulus was calculated with:

$$E = \frac{FS^3}{4WB^4}$$

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

The fracture toughness ($K_{ic}$) was obtained by calculation of this equation [5],

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

where $\eta$ is the geometrical constant (0.596), and $P$ is the indentation load on the IS beams. The geometrical constant is slightly greater than the 0.59 used by Chantikul et al. [5], because they used 2 instead of 1.854 in the Vickers equation.

**Chevron-notched beam method (CN)**

Conforming to a previous study [6], a 0.1 mm diamond-sawing disc (ISOMET 1000, Buehler, Lake Bluff, USA) was used to create a notch (Figure 3.1) with a Chevron angle $\theta$ of 60 ± 1.5° and $a_0/W$ ratio of 0.1–0.35. The beams were loaded in a three-point bending test. The simple variant of the CN was used, where just the maximum force $F_m$ is used for the calculations and no beams are rejected, regardless of the (omissible) load displacement plots, which were made at two samples per second. The Chevron angle $\theta$ and notch length $a_0$ were measured at the two fractured sections of each specimen using optical microscopy (10×, measuring precision 1 µm). The toughness was calculated with the following equation:
where $S$ is the span; $B$ the specimen width; $W$ the specimen height; $f(a_0/W)$ the stress intensity shape factor; $v$ is the Poisson’s ratio. The Poisson’s ratio was rounded to 0.25 as recommended in ISO 6872 for biaxial flexural strength calculation.

![Figure 3.1](image)

**Figure 3.1** Chevron-notched beam test.

**Single-edge-notched beam method (SENB)**

The notches of the specimens were cut with a 0.1 mm diamond saw disc. The saw depth $c$ was nearly half of the specimen's height $W$ (Figure 3.2). The specimens were fractured in a three-point bending test. The two halves of the broken samples were used for the measurement of the notch depth $c$ under an optical microscope. The length $c$ was the average of the six values at three locations of the notch: in the middle and at two lateral sides of each section. The toughness value was calculated according to the following formula [7]:

$$K_{lc} = \frac{F_c}{B} \cdot \frac{S}{W^{3/2}} \cdot f(c/W)$$

$$f(c/W) = 2.9(c/W)^{1/2} - 4.6(c/W)^{3/2} + 21.8(c/W)^{5/2} - 37.6(c/W)^{7/2} + 38.7(c/W)^{9/2}$$

where $F_c$ is the critical load; $B$ the specimen width; $S$ the supporting span; $f(c/W)$ is the stress intensity shape factor.
Statistical analysis

The significances of the material effect (difference between porcelains) and the test method effect (difference between methods) were evaluated by two-way ANOVA. The difference within each factor group was assessed by pairwise comparisons. The significance level of all statistical analysis was \( p = 0.05 \).

3.4 Results

The results are listed in Table 3.2. The data variation coefficients (data scatter), calculated as the standard deviations divided by the means (in percent), were low and ranged from 3.6 to 10.2%.

Table 3.2 Fracture toughness (MPa m\(^{-1/2}\)) of the tested dental porcelains

<table>
<thead>
<tr>
<th>Material</th>
<th>IS Mean fracture toughness±SD and the data scatter in parentheses</th>
<th>CN Mean fracture toughness±SD and the data scatter in parentheses</th>
<th>SENB Mean fracture toughness±SD and the data scatter in parentheses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duceram</td>
<td>0.884±0.088 (10.0%)</td>
<td>0.949±0.059 (6.2%)</td>
<td>0.944±0.043 (4.6%)</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>0.809±0.053 (6.5%)</td>
<td>0.843±0.062 (7.4%)</td>
<td>0.882±0.032 (3.6%)</td>
</tr>
<tr>
<td>Sintagon ZX</td>
<td>0.779±0.063 (8.1%)</td>
<td>0.698±0.045 (6.4%)</td>
<td>0.665±0.047 (7.1%)</td>
</tr>
<tr>
<td>Ducera LFC</td>
<td>0.692±0.056 (8.0%)</td>
<td>0.677±0.050 (7.4%)</td>
<td>0.708±0.072 (10.2%)</td>
</tr>
</tbody>
</table>

A total of three Duceram IS specimens were rejected because the fracture did not originate from the indentation, and extra beams were tested.

As indicated in Table 3.3, SENB displayed statistical agreement with CN for all four dental porcelains tested in this study. IS was in agreement with CN for two materials, Duceram LFC and Carrara Vincent, and with SENB only with Duceram LFC.
### Table 3.3  Pairwise comparisons of the method effect within material group

<table>
<thead>
<tr>
<th>Material</th>
<th>Method comparisons</th>
<th>Difference(ns)</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duceram</td>
<td>IS vs SENB</td>
<td>-0.060</td>
<td>0.020</td>
</tr>
<tr>
<td></td>
<td>CN vs CN</td>
<td>-0.066</td>
<td>0.011</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>IS vs SENB</td>
<td>-0.073</td>
<td>0.005</td>
</tr>
<tr>
<td></td>
<td>CN</td>
<td>(-0.034)</td>
<td>0.187</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>IS vs SENB</td>
<td>0.114</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>CN vs CN</td>
<td>0.081</td>
<td>0.002</td>
</tr>
<tr>
<td>Ducera LFC</td>
<td>IS vs SENB</td>
<td>(-0.016)</td>
<td>0.525</td>
</tr>
<tr>
<td></td>
<td>CN</td>
<td>(0.015)</td>
<td>0.550</td>
</tr>
</tbody>
</table>

### Table 3.4  Pairwise comparisons of the material effect within method group

<table>
<thead>
<tr>
<th>Material</th>
<th>Method comparisons</th>
<th>Difference(ns)</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>IS</td>
<td>Duceram vs Carrara Vincent</td>
<td>0.075</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td>Sintagon Zx</td>
<td>0.105</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.191</td>
<td>0.000</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>vs Sintagon Zx</td>
<td>(0.030)</td>
<td>0.238</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.117</td>
<td>0.000</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>vs Ducera LFC</td>
<td>0.087</td>
<td>0.001</td>
</tr>
<tr>
<td>CN</td>
<td>Duceram vs Carrara Vincent</td>
<td>0.107</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Sintagon Zx</td>
<td>0.251</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.272</td>
<td>0.000</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>vs Sintagon Zx</td>
<td>0.144</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.165</td>
<td>0.000</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>vs Ducera LFC</td>
<td>(0.021)</td>
<td>0.403</td>
</tr>
<tr>
<td>SENB</td>
<td>Duceram vs Carrara Vincent</td>
<td>0.611</td>
<td>0.021</td>
</tr>
<tr>
<td></td>
<td>Sintagon Zx</td>
<td>0.278</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.235</td>
<td>0.000</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>vs Sintagon Zx</td>
<td>0.217</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>Ducera LFC</td>
<td>0.174</td>
<td>0.000</td>
</tr>
<tr>
<td>Sintagon Zx</td>
<td>vs Ducera LFC</td>
<td>(-0.043)</td>
<td>0.096</td>
</tr>
</tbody>
</table>
In Table 3.4, all methods found differences between all materials except with Sintagon Zx; IS found no difference with Carrara Vincent, and CN and SENB found no difference between Duceram LFC and that material.

The ranking of the porcelains by the average $K_{ic}$ of all three methods from high to low is Duceram, Carrara Vincent, Sintagon Zx and Duceram LFC.

3.5 Discussion

Many fracture toughness test methods have been developed [3] and [8]: the single-edge-notched beam (SENB), and its two derivatives, the single-edge pre-cracked beam (SEPB) and the single-edge-V-notched beam (SEVNB), the Chevron-notch method (CN), surface-crack-in/by-flexure (SCF, previously also called controlled surface micro-crack), double torsion (DT), the double cantilever beam (DCB), indentation strength in/by bending (IS) with Vickers’ or Knoop's indentation, the indentation fracture method (IF), etc.

The IF, SENB and IS methods have been frequently involved in dental ceramics research [9-15]. The IF and IS, which use hardness indentations have been investigated in most reports. Other studies compared IF to SEVNB, IS to SEVNB, or IS to SCF [9-15].

As many methods often do not produce the same result for the same material, or even not give the same ranking for a set of materials [9-10, 12-15], it is clear that many methods are not always accurate. Other literature suggests that fracture toughness assessment is rather sensitive to the type of method, configuration, and processing procedures [17-24], similar to strength determination [16]. Obtained values may be inaccurate, not always consistent with each other, and different methods may offer confusing values and rankings for comparison.

For reliable and reproducible measurements of fracture toughness, recommendations of standardization organizations could be followed. At present, the single-edge pre-cracked beam (SEPB), V-notched beam (SEVNB), the surface crack in flexure (SCF) and the Chevron-notched beam (CN) have been accepted as standard methods by the ASTM or the CEN. These have been proved to give very close and reproducible $K_{ic}$ values for several advanced ceramics. The IS, IF, and SENB are not considered any further in these rather thorough standardization processes [25-29].

Many methods have shortcomings [3, 8-15, 17-29]. With most specimen configurations crack growth is unstable and $K_{ic}$ is assessed at the onset of crack extension, because at that moment the specimen breaks. Therefore, certain materials can be rather sensitive to the sharpness of the pre-crack tip, which ideally should have
the same radius as the propagating crack, which causes failure. In the SENB, the crack initiates at a not very predictable radius, related to the roughness of the notch tip, but small enough with the present materials, as the results were similar to the CN. With the SEVNB it is relatively easy to produce the V-shaped tip at the end of the notch with a razor blade. Although the microstructure of the ceramic influences the radius at the tip of the sharpened V, it is much smaller, more easily predictable, and suited to much smaller particle size materials. With the SEPB method, the specimens are pre-cracked in a specially designed bridge-anvil structure with a carefully applied high compressive load, until the slight sound of the pre-crack is heard. This should yield a quite realistic tip radius over the entire width, and some toughening (rising R-curve) may stabilize, but it is a technically sensitive method. For the SCF, it is important to locate the border of the pre-crack, which requires fractographic experience and is difficult for ceramics with porosities or a coarse crystalline microstructure. Furthermore, it is critical to remove the residual stress around the Knoop's indentation. The amount of removal, however, varies across materials.

The CN method has been designed to overcome the problems with elaborate pre-cracking. The triangularly widening crack front leads to insensitivity to crack furcation and origin deviation [3, 8, 25-31]. With the CN notch, pre-cracking occurs automatically and inevitably when the load is applied. Already at a small load, a crack initiates in the tip of the triangle, due to stress concentration. At first the crack should grow stably with increasing load, because the crack front widens. This was visible as bending of the load displacement plots. The formula calculates $K_{ic}$ at the point where the maximum load occurs, which is at the crack length where further crack propagation may become unstable and the specimen breaks. Most specimens fractured beyond the maxima of the load displacement plots, where the load had been constant or decreasing a few percent for several seconds, which indicates stable crack growth. With each material two or three beams fractured before, or shortly before, the plots had fully bent horizontally. The $K_{ic}$ of one Duceram CN beam was 3% overestimated as it showed a small pop-in, i.e. the load increased linearly to a sharp peak, where it dropped about 4% and increased more slowly to a normally rounded peak of about 3% less. This beam's $K_{ic}$ was 7% greater than the Duceram average (Table 3.2). One LCF CN beam showed a single pop-in, i.e. it fractured with a single linear peak, suggesting unstable cracking. This beam's $K_{ic}$ was 17% greater than its average. With 10 specimens tested, these pop-ins have increased the averages by 0.3 and 1.7%, respectively. It seems safe to use just the maximum force with the CN method.
Although only a large notch is sawn, the pre-crack at which $K_{IC}$ is assessed, is generally a growing crack. This method could rival the SEPB, but the crack front is less wide. Because the CN is reportedly elaborate, it should perhaps be stated that it is not when the beams are prepared one by one as in the present study. Compared to the simple SENB each specimen needs to be rotated and sawn again, which is perhaps less effort than the IS specimens, as these require extra ($E$ and $H$) experiments and specimens. The two broken halves of a long rod CN experiment may be used for two short-rod experiments, when small amounts of experimental material are available. In dental literature, one article [32] used the short-rod CN method to measure the fracture toughness of three dental ceramics.

Because dental porcelains and ceramics have a broad range in microstructure, chemistry, and fracture toughness [33], it could be useful to systematically assess the comparability of the various test methods with the standardized (ASTM, CEN) methods, since these have been proved to work on advanced ceramics.

The IS method seems an economically affordable alternative to $K_{IC}$ estimation only when a diamond saw is not available, as its limitations are much more complex than the straightforward notch problem of the SENB.

Considering its history, the Chevron-notched beam values of the present study could perhaps be seen as more accurate than the others. With the present four dental porcelains only the SENB method was statistically comparable to the CN.

3.6 Conclusion

1. The CN method, and recording just the highest load seems accurate with dental ceramics. This ATSM standard requires less effort than many other tests.
2. With the present materials the SENB was statistically close to CN.
3. IS was not always in statistical agreement with SENB or CN and further investigation is needed, as the limitations are unclear.
4. The fracture toughness sequence from high to low is Duceram, Carrara Vincent, Sintagon Zx, and Duceram LFC, according to the three test methods.

3.7 References

Fracture toughness of three test methods


