Strength testing variables in dental ceramics

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CHAPTER 2

Influence of Test Methods on Fracture Toughness of a Dental Porcelain and a Soda Lime Glass

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

Objective: The aim of this study was to investigate the influence of the test method on fracture toughness of a dental porcelain and a soda lime glass.

Materials and methods: Three methods were used to determine fracture toughness: the indentation strength (IS) by bending, chevron-notched beam (CNB), and the single-edge-notched beam (SENB). In the IS method, the ratio of elastic modulus to hardness \((E/H)\) in the formula was determined by two methods: individual measurement for \(E\) and \(H\) (ISM) as well as direct estimation from Knoop's indentation method (ISK). The tested materials were a dentin porcelain, a traditional feldspar-based leucite-reinforced glass ceramic (Carrara Vincent), and a soda lime glass.

Result: Carrara Vincent showed a higher toughness \((P<0.01)\) than glass with all three test methods. The toughness values manifested significant differences between the methods used \((P<0.01)\). The two-way analysis of variance suggested that the materials tested and the test methods used had interaction effects, which statistically means that differences in materials and methods influenced the comparability of the toughness result. In this study, a first step was made to compare different toughness test methods by testing the toughness of a traditional feldspar-based leucite-reinforced glass ceramic and a soda lime glass that has a homogeneous microstructure.

Conclusion: An interaction effect of the method and the material used was shown. As a consequence, none of the methods tested is suitable as a universal fracture toughness test method. Further research is needed to investigate more extensively the influence of material composition on the fracture toughness test methods' comparability.
2.2 Introduction

Strength is commonly seen as an important parameter for understanding the clinical performance of dental ceramic restorations as it reflects an important mechanical property. Since the tensile strength of ceramics is much lower than the compressive strength, ceramic restorations often fail in areas of tensile stress. The traditional method for restoration design and material selection is based on attempts to reduce the tensile stresses generated in the structure under load and to select materials with greater strengths than the expected applied stresses [1-4].

Unfortunately, with extremely brittle materials such as ceramics, high strength does not imply a satisfactory fracture resistance [2-4]. Fracture is caused by a propagating crack, which often originates from flaws and extends when the applied stress exceeds a certain threshold. In very brittle materials, this threshold largely depends on the crack tip radius, flaw size, flaw distribution, and fracture toughness.

Fracture toughness is one of the most important material properties in fracture mechanics for brittle materials and is assumed to be independent of flaw size, specimen shape, and the stress concentration acting on the surface. Fracture toughness ($K_{lc}$) of a brittle material is characterized by a critical level of the stress intensity factor near the crack tip at which a crack will start to propagate. For ceramics that have a primary disadvantage of brittleness and contain many flaws, fracture toughness is, therefore, more elucidating than strength. Obviously, the availability of an accurate method for fracture toughness determination is very important. In the dental literature, greater attention is being drawn to fracture toughness measurements, resulting in an increasing number of publications [5-12].

Various test techniques have been developed for the determination of fracture toughness: the single-edge-notched beam (SENB, by three- or four-point bending) [3, 4, 12] and its derivatives single-edge-precrack-notched beam (SEPB/SEPNB) [4, 7, 13] and single-edge-V-notched beam (SEVNB), [5, 6] chevron-notched (CN) specimen (short rod/bar, three/four-point bending long beam/rod), [8,13-15] double-torsion, double-cantilever beam (DCB), indentation strength (IS) by bending with Vickers or Knoop indentsations, [12, 16] indentation fracture (IF) method, [6, 12, 17] and surface-crack-by-flexure (SCF, also called controlled surface microcrack).[11] In dental ceramics research, IF, SENB, and IS methods have frequently been used for the ranking of fracture toughness. The CN method, which rarely appears in the dental literature for ceramic evaluation, was developed in the late 1970s. It is different from SENB in that the designed crack for extension has the shape of a triangle instead of a rectangle.
In spite of the fact that fracture toughness is a material property and therefore the test method to determine this property should not affect the value, it might be expected that just like strength variation attributed to the difference of test techniques, [18] fracture toughness values might be sensitive to and might be affected by the testing and processing methods used.

The aim of this study was to determine the influence of test methods on fracture toughness data of dental ceramics. In this study, fracture toughness values of a dental porcelain and a soda lime glass were determined using three different methods.

3.3 Materials and Methods

Specimen Preparation

Three fracture toughness tests were selected for the study: the IS method, the CN beam (CNB) method, and the SENB method. For each method, at least 10 beam-shaped specimens were prepared (Table 2.1). Carrara Vincent porcelain (Elephant Dental B.V., Hoorn, the Netherlands) is a dental dentin porcelain for metal and all-ceramic applications; a soda lime glass was used as reference. Green bodies of porcelain powder were formed in a metal fixture and were then fired in a dental porcelain oven (STRATOS, Elephant Dental B.V.) according to the manufacturer's instructions, except that the cooling time was extended to 1 h to prevent breakage of the baked bodies caused by fast cooling, because their size and thickness were much greater than that of dental ceramic restorations. After firing, the porcelain bodies were ground to a thickness of 3.00±0.05 mm in a grinding device (VEM Metallurgy, Vos & Van Eijk Metallurgie B.V., Houten, the Netherlands) with 30 /µm diamond pastes. Subsequently, the bodies were cut into bars with a 0.5 mm diamond saw (ISOMET 1000, Buehler Ltd, Lake Bluff, IL).

All IS beams followed a series of wet grinding and polishing procedures (ECOMET Grinder/Polisher, Buehler Ltd, Evanston, IL) with silicon carbide paper (400, 600, and 1200 grit), during which the sharp edges of rectangular specimens were blunted.

Annealing treatments of specimens were carried out to remove residual stresses. Annealing temperatures were held at 450°C for 1.5 h. Then, specimens were cooled to 100°C in the furnace for another 1.5 h. The annealing temperature was close to the glass transition temperature of the Carrara Vincent porcelain.
Soda–lime–silica glass beams were cut from glass plates and were prepared according to the same processing method to obtain comparable sizes and surface roughness.

**Table 2.1** The test configuration of three measurement techniques

<table>
<thead>
<tr>
<th>Test method</th>
<th>CNB</th>
<th>SENB</th>
<th>IS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Precrack technique</td>
<td>Notch</td>
<td>Notch</td>
<td>Micro-crack</td>
</tr>
<tr>
<td>Precrack type</td>
<td>Large crack</td>
<td>Large crack</td>
<td>Small crack</td>
</tr>
<tr>
<td>Precrack processing method</td>
<td>sawing</td>
<td>sawing</td>
<td>Vickers indentation</td>
</tr>
<tr>
<td>Fracture plane</td>
<td>Chevron triangle</td>
<td>Rectangle through half section</td>
<td>Rectangle through whole section</td>
</tr>
<tr>
<td>Load mode</td>
<td>3-point bending</td>
<td>3-point bending</td>
<td>3-point bending</td>
</tr>
<tr>
<td>Load speed</td>
<td>0.05 mm/min</td>
<td>0.05 mm/min</td>
<td>0.05 mm/min</td>
</tr>
<tr>
<td>Specimen dimension proportion(d:b:S)</td>
<td>2 : 3 : 12</td>
<td>1 : 2 : 8</td>
<td>2 : 3 : 22</td>
</tr>
<tr>
<td>Dimension (d:b:S) for porcelain</td>
<td>3 × 4.5 × 26 mm</td>
<td>3 × 6 × 26 mm</td>
<td>2 × 3 × 25 mm</td>
</tr>
<tr>
<td>Dimension (d:b:S) for glass</td>
<td>3.8 × 5.7 × 30 mm</td>
<td>3.8 × 7.6 × 50 mm</td>
<td>3.8 × 4.5 × 50 mm with span of 40 mm</td>
</tr>
</tbody>
</table>

**IS Based on the Bending Method**

Vickers hardness indentations were made (HM-124 Hardness Testing Machine, Mitutoyo Corp., Kanagawa, Japan) on the tensile surface in the middle of the beams at a load of 9.8 N for glass and 19.6 N for dental porcelain. Variation in the indentation load was introduced to make the ratio of crack length to indentation diagonal equal to about 2.0. Within 0.5 h following the indentation, the beam was loaded in a tensilometer (Instron Universal Mechanical test machine, Instron Corp., Hy Wycombe, U.K.) on the side opposite the indentation in three-point bending until fracture occurred. Specimens in which the fracture did not originate from the indentation notch were excluded; therefore, testing was continued until at least 10 acceptable specimens were acquired.

The fracture strength of indented specimens (IS) was calculated according to the following formula:
where $\sigma_f$ is the bending strength, $W$ is the fracture load, $L$ is the span length, $b$ is the specimen width, and $d$ is the specimen thickness. The fracture toughness values were then calculated using the following formula [16]:

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

(2)

where $\eta$ is a geometrical constant (0.59), $E$ is the elastic modulus, $H$ is the Vickers hardness, $\sigma_f$ is the IS, and $P$ is the indentation load.

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

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

(3)

The Vickers hardness was measured on broken specimens ($n=10$) using a load of 1.96 N for 15 s. The hardness was calculated using $H=1.854P/(2l)^2$, where $P$ is the indentation load (1.96 N), and $l$ is half of the average length of two diagonals of the indentation measured with a precision of 0.1 $\mu$m. The ratio of elastic modulus to hardness ($E/H$) was determined in two different ways: first, $E$ and $H$ were measured individually as described above and their ratios $(E/H)_M$ were calculated; second, the ratio was estimated from the length/width ratio of a Knoop indentation, with $y'/x' = y/x - \alpha/(E/H)_K$, where $x$ and $y$ are defined by the Knoop indenter geometry, $y/x = 7.11$. $x'$ and $y'$ are the measured length of long and short diagonals of Knoop indentation; $\alpha$ is the fitting gradient and amounts to 0.45 [19].

The $(E/H)_M$ and $(E/H)_K$ ratios were used in the formula for IS fracture toughness and led to two toughness results: IS$_M$ and IS$_K$, respectively.

**CNB Method**

Three-point bending was performed on the CN specimens according to previous research on the CNB test technique for plane-strain fracture toughness of brittle
Influence of Test Methods on Fracture Toughness

materials [20] (see Figure 2.1). A chevron notch was made with a 0.1 mm diamond sawing disk (ISOMET 1000, Buehler Ltd), which yielded to a chevron angle \( \theta \) of \( 60^\circ \pm 1.5^\circ \) and a pre-crack ratio \( (r_0/b) \) of 0.1–0.35. As with the traditional three-point bending test, the load was recorded during the experimental procedure, and the maximum load \( W \) was obtained from this record. The Chevron angle \( \theta \) and Chevron precrack length \( r_0 \) were measured on the two fractured sections of each specimen by optical microscopy (× 10, measuring precision 1 \( \mu \)m). The fracture toughness was calculated according to the following equation:

\[
K_{ic} = \frac{W}{2d^{3/2}(1-v^2)^{1/2} \tan^{1/2}(\theta/2)} \cdot f(a_0/d)
\]

(4)

where the Poisson's ratio \( v \) was assumed to be 0.25, as recommended by ISO 6872 for the biaxial flexural strength calculation, and \( f(r_0/b) \) is the stress intensity shape factor. The actual values of \( r_0 \) and \( b \) of each specimen were used.

**Figure 2.1** Chevron-notched beam test.

**SEN'B Method**

For the SEN'B test, the notch of a specimen was machined with the 0.1 mm thick diamond saw disk. The saw depth, \( a \), was nearly half that of the specimen's width, \( b \) (see Figure 2.2). The specimens were fractured in a three-point bending fixture. The two halves of the broken specimens were used for notch depth measurement under an optical microscope (magnification × 10, reading precision 1 \( \mu \)m). The depth, \( a \), was the average of six values at three locations of the notch: middle point and two sides of each section. The fracture toughness value was calculated according to the following formula [21]:

\[
f(a_0/d) = 17.959 + 20.708(a_0/d) + 179.53(a_0/d)^2
\]
\[ K_c = \frac{W L}{b d^{3/2}} \cdot f(a/d) \quad (5) \]

\[ f(a/d) = 2.9(a/d)^{1/2} - 4.6(a/d)^{3/2} + 21.8(a/d)^{5/2} - 37.6(a/d)^{7/2} + 38.7(a/d)^{9/2} \]

where \( \sigma_f \) is the fracture stress, \( W \) is the critical load, \( d \) is the specimen thickness, \( b \) is the specimen width, \( L \) is the span length between supports, \( a \) is the notch depth, and \( f(a/b) \) is the stress intensity shape factor. The actual values of \( a \) and \( b \) of each specimen were used. For brittle materials, \( F_c \) is equivalent to the maximum load.

Figure 2.2  Single-edged-notched-beam test.

Statistical Analysis

Two-way analysis of variance was utilized for determining the significance of the material effect, the test method effect, and the interaction between these effects. The difference within each material group was analyzed by pairwise comparisons of tests for a simple method factor within the other factor–material group. The test methods were assigned to three groups comprising combination 1 of IS\(_M\), CNB, and SENB, combination 2 of IS\(_K\), CNB, and SENB, and a paired combination of IS\(_M\) and IS\(_K\). All statistical analyses were assessed at an \( \alpha \) level of 0.05.

2.4 Results

The fracture toughness results for the tested materials are listed in Table 2.2. The statistical analysis results are presented in Tables 2.3, 2.4, and 2.5. In all cases, data scatter was low, as the standard deviations ranged from 2.5% to 7.4% of the mean values.

The main material effect and the main method effect as well as the material by method interaction were significant \( (P \leq 0.05) \) for all method combinations. The traditional leucite-reinforced feldspar-based glass-ceramic Carrara Vincent was slightly tougher than glass, which was demonstrated with each of the three test methods. At the same time, for each material, the toughness results differed slightly by
the test method. Moreover, the differences between the materials were also influenced by the methods used.

**Table 2.2** Fracture toughness (in MPa m$^{-1/2}$) of five materials with four test methods

<table>
<thead>
<tr>
<th>Materials</th>
<th>$K_{IC}$ (average, standard deviation, and scatter in percent)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$IS_M$</td>
</tr>
<tr>
<td>Glass</td>
<td>0.77±0.02(2.5%)</td>
</tr>
<tr>
<td>Carrara Vincent</td>
<td>0.82±0.05(6.5%)</td>
</tr>
</tbody>
</table>

$K_{IC}$=Fracture toughness of mode I crack, $IS_K$=Indentation strength method using $(E/H)_K$, $IS_M$ = Indentation strength method using $(E/H)_M$, CNB=Chevron notched beam method, SENB=Single edge notched beam method.

**Table 2.3** Tests of between-Subjects Effects

<table>
<thead>
<tr>
<th>Subjects combination</th>
<th>Effect source</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>$IS_M$ and $IS_K$</td>
<td>material</td>
<td>9.827</td>
<td>0.005</td>
</tr>
<tr>
<td></td>
<td>method</td>
<td>2508.939</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>material-method interaction</td>
<td>876.556</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>$IS_M$, SENB and CNB</td>
<td>material</td>
<td>91.868</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>method</td>
<td>8.069</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td>material-method interaction</td>
<td>7.326</td>
<td>0.001</td>
</tr>
<tr>
<td>$IS_K$, SENB and CNB</td>
<td>material</td>
<td>95.649</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>method</td>
<td>7.917</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td>material-method interaction</td>
<td>6.139</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Although mean toughness values for $IS_M$ and for $IS_K$ were very close together for both materials, the differences between means was statistically significant. The difference for glass (0.002) was somewhat smaller when compared with that for Carrara Vincent (0.009). Meanwhile, for both materials, SENB indicated a greater fracture toughness than CNB. However, statistically, for glass, both $IS_M$ and $IS_K$ gave toughness values comparable to that for SENB, and higher than that for CNB, whereas for Carrara Vincent, the mean values were comparable to that for CNB but lower than that for SENB.
### Table 2.4  Tests for simple method within material effects

<table>
<thead>
<tr>
<th>Subjects combination</th>
<th>Material</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>IS(_M) and IS(_K)</td>
<td>glass</td>
<td>219.304</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>Carrara Vincent</td>
<td>3043.405</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>IS(_M), SENB and CNB</td>
<td>glass</td>
<td>8.959</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>Carrara Vincent</td>
<td>6.394</td>
<td>0.003</td>
</tr>
<tr>
<td>IS(_K), SENB and CNB</td>
<td>glass</td>
<td>9.289</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>Carrara Vincent</td>
<td>4.779</td>
<td>0.012</td>
</tr>
</tbody>
</table>

### Table 2.5  Pairwise comparisons with regard to toughness as dependent variable

<table>
<thead>
<tr>
<th>Subjects combination</th>
<th>Material</th>
<th>Method comparisons</th>
<th>Difference</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>IS(_M), SENB and CNB</td>
<td>glass</td>
<td>IS(_M) vs SENB</td>
<td>0.002</td>
<td>0.905</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CNB vs SENB</td>
<td>0.069</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SENB vs CNB</td>
<td>0.067</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td>Carrara Vincent</td>
<td>IS(_M) vs SENB</td>
<td>-0.066</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CNB vs SENB</td>
<td>-0.026</td>
<td>0.163</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SENB vs CNB</td>
<td>0.040</td>
<td>0.040</td>
</tr>
<tr>
<td>IS(_K), SENB and CNB</td>
<td>glass</td>
<td>IS(_K) vs SENB</td>
<td>0.005</td>
<td>0.798</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CNB vs SENB</td>
<td>0.072</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SENB vs CNB</td>
<td>0.067</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td>Carrara Vincent</td>
<td>IS(_K) vs SENB</td>
<td>-0.056</td>
<td>0.004</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CNB vs SENB</td>
<td>-0.016</td>
<td>0.379</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SENB vs CNB</td>
<td>0.040</td>
<td>0.041</td>
</tr>
</tbody>
</table>

#### 2.5 Discussion

The pronounced effect of the materials on the fracture toughness can be explained by their microstructure. Soda–lime–silica glass is microstructurally homogeneous and devoid of crystals. Glass was chosen as a control since it is believed to be a good counterpart for ceramic materials because of its brittleness and toughness, which is usually 0.77 Mpa m\(^{-1/2}\) [22]. The leucite in the traditional leucite-reinforced feldspar-based glass ceramic Carrara Vincent may not only have a reinforcing effect but may also have a toughening effect, while the chemical composition of the matrix may obviously contribute to its toughness increase in any toughness test by deflecting crack extension [5, 7].
It is well-known that for proper material evaluation and development, the critical stress intensity factor, $K_{IC}$, better known as fracture toughness, should be properly obtained. At present and historically, the IF method, the IS by bending method, the SENB and its derivates SEVNB and SEPB, and SCF methods have been applied in the fracture toughness testing of dental ceramics.

SENB is a traditional method, established to measure straightforward fracture toughness, and has often worked as a reference or standard test technique [13, 23-28]. Values obtained from this test proved to be valid only for ceramics containing coarse crystals (20–40 μm). For ceramics containing fine crystals, less than 2–4 μm in size, the values obtained are expected to be overestimated. In most instances, the notch tip width is usually and practically 100 μm (as in this study) or wider, which tends to increase the fracture toughness measured on finer crystalline materials. There are two modifications based on this straight-through notch technique. Single-edge-precrack (notched) beam (SEPNB, or SEPB) specimens have a fatigue-induced crack from the tip of the notch and often serve as a reliable reference method in comparison of toughness measurement tests [7, 13, 25, 27]. The SEVNB was developed to overcome the shortcoming of a sharp crack front in SEPB. The saw-cut notch is sharpened with a razor blade and oil-lubricated diamond paste in order to reduce the notch width to twice the grain size [5-7, 23-25, 27]. Both derivatives are time and technique consuming and the pre-cracking/V-notching process easily confers pre-crack front aberrations or sawing-generated penny-shaped flaws, which can lead to a discrepancy in the measured toughness [5]. The SEPB and SEVNB techniques consume more material than the CN specimens, which is a disadvantage as newly developed materials are usually provided in very small quantities. Another problem is that the beam configuration tends to be unstable because most test machines have difficulties in achieving stable crack growth in brittle SENB specimens [13].

In dental journals, only one report was based on the use of the CN method for dental ceramics [8]. This technique is particularly suitable for very brittle materials such as ceramics. The CN toughness is based on plane strain rather than the straight-through-crack theory [13-15, 24, 25, 27]. Because of the high stress concentration at the tip of the chevron notch, a crack initiates at a low applied load. When the crack propagates, the stress intensity factor decreases to a minimum, because the crack front becomes wider because of the triangular shape of the notch. At the minimum value, the crack grows stably with increasing load. Above this point, the crack propagation becomes unstable and the specimen snaps. At the point of minimum stress intensity factor, the fracture toughness can be evaluated with the maximum test load.
An important advantage is that pre-cracking of the specimen is not necessary as this occurs during the first and stable part of the test before the maximum load is reached. Moreover, this is a way of creating a pre-crack with a tip, which resembles that of a propagating crack as much as possible, at least theoretically.

As pointed out in the literature, [14, 15] the calculation of fracture toughness should take the stress intensity factor (instead of using only the maximum fracture value) and the load–line displacement as a function of crack length (generally \( a_0/W \)) into account in order to obtain compliance calibrations to verify that the \( R \) curve has become horizontal and to some extent compensate when it has not. In addition to introduction of Poisson's ratio into the formula, this may engender the comparability of results from a CN specimen with other results [3, 14, 15]. With regard to this consideration, the assumption of 0.25 for the Poisson's ratio of the materials tested in this study might cause a small variance from reality. However, when \( \nu \) in the formulas is changed to 0.3 or to 0.2, \( K_{lc} \) changes to +1.50% or −1.18%, respectively. This discrepancy is very low in consideration of data scatter.

SCF is a good method to assess the fracture toughness, with the advantage of simulating a real surface flaw, the size of which is required in the calculation of the fracture toughness [3, 10, 11, 29, 30]. The flaws are often induced by a Knoop indentation rather than a Vickers indentation. With the help of fractographic analysis, this indentation-induced flaw is demarcated after a three- or four-point flexural specimen is fractured through this artificial flaw on the tensile side. A disadvantage is that the flaw identification requires experience and knowledge, and might be affected by subjective judgment [11, 29]. The residual stress and lateral cracks caused by the indentation interfere with the true value of SCF fracture toughness tests by influencing the maximum load at fracture [30]. Unfortunately, the exact amount of material removed from the residual stress zone and lateral cracks is not well-known at present, and requires detailed and individual investigation. Such an interference is assumed to exist in most dental ceramics since this phenomenon appears in brittle materials with toughnesses less than 3 MPa·m\(^{1/2}\) [30]. For materials with evident porosity, coarse grain, and ready generation of lateral cracks, SCF is not preferred because of the difficulty in delineating the artificial flaw and its size, and the necessity that fracture of all valid samples tested must originate from that artificial flaw/indentation.

Introduced by Chantiful et al. (1981), the IS method is a convenient method that does not require preparation of a macrocrack (i.e., notch); instead, a small crack (microcrack) is induced in the tensile surface in the form of a Vickers indentation, similar to the technique used for the SCF method [3, 5, 9, 11, 12, 16, 28]. Moreover, it
is unnecessary to gauge the produced crack size for calculation of the fracture toughness, which only requires the indentation load, and as described, the post-indentation crack propagation does not affect the test result very strongly [16]. Nevertheless, increasing the time interval between indentation and the bending test may contribute to data scatter, and lateral cracks generated by the Vickers indentation, especially at a higher load, may cause inaccuracy [5]. Subcritical crack growth of ceramics is believed to be associated with residual stress and moisture. Thus, use of the same criteria for the timeframe from indentation to fracture and oil coverage or an inert gas atmosphere at the indentation site should be useful. Annealing of the specimens before indentation may also be helpful in reducing the error [5, 28] but may be inconsistent with clinical practice. As a small crack, the indentation-induced crack in IS specimens has to be sufficiently distinct from the intrinsic flaws within the specimens. Similar to the SCF test, when the fracture initiates at a location other than the artificial flaw, the test results become invalid. Conceivably, changing the test methods such as three-/four-point, biaxial bending, etc., might cause a greater discrepancy in the calculated toughness value [9, 18] This simple method needs more study for general usage and recommendations for standard organizations.

All fracture toughness methods inevitably face the influence of a material's $R$-curve behavior [2, 3, 5, 11, 13-15]. Although a rising $R$ curve reflects a generally favorable material property, it also renders any toughness test invalid. During the first part of crack propagation with such materials, the toughness increases, while for the tests it should have reached its steady-state value, which is believed to be size independent.

Also, in this respect, the methods are divided into two types: long, large-crack, or macro-crack toughness methods, and short, small-crack, or microcrack toughness methods. The former includes SENB, SEPB, SEVNB, and CN tests, and the latter type is comprised of SCF and IS tests. Although the fracture toughness of a rising $R$-curve ceramic is no longer a material constant, results from large- and small-crack test methods represent different performances of the materials' resistance to crack propagation under specific service conditions [2, 4]. In other words, methods could be chosen according to service conditions of the materials being tested.

By reviewing the weaknesses of each test method and individual test method difficulties, one can better understand the results of this study, especially the material–method interaction and method effect on the fracture toughness, which can be influenced by many factors. As reported in the literature, the results of individual fracture toughness tests may or may not be comparable to the results of other
investigators [5-7, 23-29]. The fracture toughness can be reported as apparent fracture toughness. Although the outcome of this study revealed differences by material and test technique used, the goal in refining $K_{IC}$ test methods is to achieve consistent, repeatable results. A few investigations have been performed in an effort to demonstrate that CNB, SEPB, and SCF techniques are in good agreement with each other when using an updated revision of the stress-intensity shape factor and elimination of bias factors [30-33]. With the V-notch technique, other researchers have also attempted to improve the comparability of toughness data [27]. The statistical results of this study imply that in practice, fracture toughness tests always require extremely careful processing of the notch or indentation techniques.

2.6 Conclusion

In this study, a first step was made to compare different toughness test methods by testing the toughness of a traditional feldspar-based leucite-reinforced glass ceramic and a soda lime glass that has a homogeneous microstructure. An interaction effect of the method and the material used was shown. As a consequence, none of the methods tested is suitable as a universal fracture toughness test method. Further research is needed to investigate more extensively the influence of material composition on the fracture toughness test methods' comparability.

2.7 References


