This study compared the fracture toughness values (K_{IC}), which were derived from simplified techniques: the indentation fracture (IF), the indentation strength (IS), and fractographic approach to that from a standard testing using surface cracks in flexure (SCF). Forty bar specimens, twenty IPS Empress® Ceram were prepared. Ten specimens in each material were tested by IF technique, IS technique and fractographic approach, and additional 10 specimens were tested by the SCF technique. This study showed that the mean K_{IC} derived from fractographic approach were not significantly different from that of the SCF in both materials (p>0.05) whereas the mean K_{IC} from indentation techniques rarely agreed with those of the standard technique. The K_{IC} determination is sensitive to the methods used that affect accuracy. Consequently, test selection should be based on a sound understanding and inherent limitations of each technique.

**Keywords:** Fracture toughness, Fractography, Indentation, Knoop, Vickers

# INTRODUCTION

Since dental ceramics have been widely used for fixed dental prostheses (FDPs) that support the occlusal force, it is essential for these brittle materials to withstand such loading or stress during function. The common approach for reporting this ability is the material’s strength. However, although strength is a specimen-dependent material property, it is not an intrinsic property of material because it is dependent on crack size. Moreover, using strength to estimate material performance is meaningful when we have insightful knowledge of factors such as: microstructure, processing history, testing methodology and environment, and flaw distribution\(^1\). For dental ceramics, it is inevitable that defects such as surface flaws, foreign inclusions, or agglomerates are introduced into the materials during processing. These strength-controlling flaws influence the performance or failure strength of these brittle materials. In most fracture tests, critical crack size of the failure origin is not measured or controlled\(^5\). In contrast, fracture toughness (K_{IC}) is a property that is a measure of a material’s resistance to brittle fracture when a crack is present\(^9\). It is an intrinsic property of ceramics, which indicates the ability of ceramics to resist crack extension and associated catastrophic failure\(^4\). Fracture toughness is generally independent of crack size. Various techniques have been employed to determine the fracture toughness, which are classified as large and small crack techniques\(^2\). Large crack techniques include a double-cantilever-beam type specimen, single-edge notched beam, and the chevron-notch flexure beam designs. Examples for small crack techniques are usually based on a flexure test using a controlled crack or fractography based on a natural flaw.

One of difficulties for fracture toughness tests is the difficulty in accurately measuring the K_{IC} value\(^7\). Criteria for standardized tests were recommended in order that they can provide good estimations of fracture toughness\(^9\). The tests that meet criteria for a standard test, for example, the chevron notch beam (CNB), the single edge precracked beam (SEPB), the surface crack in flexure (SCF), the short chevron-notched, the double cantilever beam and the single edge V-notch beam (SEVNB). ASTM recommends some standard test designs for advanced ceramics such as the chevron-notched beam, the precrack beam specimen and the surface-crack in flexure\(^9\). Nevertheless, these tests vary in their difficulty in execution, and the high cost and difficulty of specimen preparation.

The crack indentation technique that requires pyramidal hardness indenter has been widely used to determine mechanical properties of brittle materials such as hardness, brittleness and fracture toughness. In addition, it has also been used to estimate residual stress based on the difference between the crack length of stressed and unstressed materials\(^10\). The indentation technique was first used as a fracture toughness test since the 1970s\(^12\). The advantage of the technique is that it requires a small sample for rapid evaluation of mechanical properties and it may also be useful for material development or quality control\(^10\). However, the Vickers indentation technique has been criticized as an unreliable method for calculating the fracture toughness or any other fracture resistance parameter, and even for a comparative reason\(^5\). It can only be used for a first rough estimation for fracture toughness because most calibration constants are empirically determined and adjusted until a reasonable value is achieved according to the materials under investigation\(^10\). Nevertheless, this approach still is a
popular method for testing mechanical properties of ceramic materials because of its expediency, ease of use, and small sample geometry\textsuperscript{10}. The most common techniques include the indentation fracture (IF) and the indentation strength (IS) methods\textsuperscript{6,14,15,16}. Quantitative fracture surface analysis employs fracture mechanics principles to characterize features on the fracture surface. It is often used in failure analysis to identify the fracture origin and to characterize specific aspects of failure\textsuperscript{10}. It provides key information on mechanical strength parameters, and it has been employed to analyze clinically failed prostheses\textsuperscript{17-20}. Fractography, combined with a controlled flaw produced by a Vickers hardness indenter and a conventional flexure condition, has been used as a simple technique to investigate the fracture toughness of dental ceramics or other brittle materials\textsuperscript{2,5,21,22}. This approach is similar to SCF method that is the standard technique recommended by ASTM for the fracture toughness test of advanced ceramics\textsuperscript{9}. Therefore, the objective of this study was to compare the fracture toughness tests of two dental ceramics that are derived from an indentation technique and the fractographic approach, with a standard method. In addition, the null hypothesis of this study was that the fracture toughness values deriving from the indentation techniques and fractographic approach were not different from that of a standard technique, and the surface crack in flexure.

**MATERIALS AND METHODS**

Two dental ceramics (IPS e.max\textsuperscript{®} Ceram and IPS Empress\textsuperscript{®} Esthetic) were used in this study. Composition, firing temperature, and mechanical properties of the materials are shown in Table 1.

**Specimen preparation**

Twenty bar specimens of two commercial dental ceramics were prepared as follows:

- IPS e.max\textsuperscript{®} Ceram (Ivoclar-Vivadent, Schaan, Liechtenstein) —Twenty glass bars were fabricated by the powder condensation method. The powder was mixed with the corresponding liquid to a slurry consistency, and condensed in an aluminum mold (6×8×35 mm). A 10 kg load was applied to the condensed mixture. The oversized mold was used to compensate for subsequent sintering shrinkage. The condensed bars were sintered according to the manufacturer’s instructions and slowly cooled to room temperature. All bar specimens were ground and finished to produce final dimensions of 3×4×25 mm by using silicon carbide abrasive paper (240, 400, 800, 1200 and 2000 grade consecutively) and a 1-µm diamond paste until a mirror-like surface was achieved.

- IPS Empress\textsuperscript{®} Esthetic (Ivoclar-Vivadent) —Twenty bars were fabricated by hot isostatic pressing and the lost-wax technique. Slightly enlarged plastic patterns were sprued and invested in the silicone mold. After the investment was completely set, the investment mold was heated in a programmable furnace at 850°C. Subsequently, the ceramic was hot-pressed into the mold according to manufacturer’s instruction. Specimens were cleaned, finished and polished to achieve the same dimensions as used for the IPS e.max\textsuperscript{®} Ceram bars.

Before testing, all the specimens were annealed at 570°C and 690°C for IPS e.max\textsuperscript{®} Ceram and IPS Empress\textsuperscript{®} Esthetic, respectively (approximately 75°C above the glass transition temperature [Tg]) in order to eliminate all residual stresses before testing. The Tg of both materials was derived from the manufacturer’s technical data and from another study\textsuperscript{23}. The specimens were cooled slowly from the annealing temperature to room temperature over a period of 30 min.

**Fracture toughness test**

All tests were performed under ambient conditions, and four methods were used for the fracture toughness tests.

- Group I: Vickers indentation fracture (VIF) —Ten specimens of each ceramic were indented using a Vickers hardness indenter; each indentation produced median-radial crack (semicircular or semieliptical crack) in the middle of polished surface. The optimal load for each material was determined by comparing the extending crack length (c) which measured from the center of indentation to the length of the half-diagonal pyramidal indenter (a); a load must be great enough to produce a ratio of c/a that is greater than 2.3 times of half diagonal pyramidal length without severe damage\textsuperscript{24}. For this reason, 9.8 N and 19.6 N loads were used for IPS e.max\textsuperscript{®} Ceram and IPS Empress\textsuperscript{®} Esthetic, respectively. The crack length of each specimen was measured using

| Table 1 The composition, firing temperature and mechanical properties of materials used in this study according to the manufacturer |
|---------------------------------------------------------------|---------------|-----------------|-----------------|-----------------|------------------|
| **Materials** | **Manufacturer** | **Composition** | **Firing temperature (°C)** | **Vickers hardness (MPa)** | **Biaxial strength (MPa)** | **Fracture toughness (MPa·m\textsuperscript{1/2})** |
| IPS Empress\textsuperscript{®} Esthetic | Ivoclar Vivadent AG, Schaan, Liechtenstein | Leucite-reinforced glass | 1,025 | 6,200 | 160±8 | 1.3 |
| IPS e.max\textsuperscript{®} Ceram | Ivoclar Vivadent AG | Fluoroapatite glass | 760 | 5,400 | 90±10 | — |
optical light microscope at a magnification of 100x to 200x one hour after indentation, and the fracture toughness (KIC) was calculated from equation (1).

$$K_{IC} = 0.016 \left( \frac{E}{H} \right)^{1/2} \left( \frac{P}{c^{3/2}} \right)$$  \hspace{1cm} (1)$$

where: $K_{IC}$ is the critical stress intensity factor or fracture toughness of material (MPa$\cdot$m$^{1/2}$); $E$ is elastic modulus; $H$ is hardness; $P$ is the indentation load (N); and $c$ is the crack length measured from the center of the indentation (m).

Group II: Indentation strength —After the indentation crack were produced and the crack lengths were measured, the 10 bar specimens from group I were tested by four-point flexure (with a 20-mm support span length and 10-mm loading span length) until fracture occurred using a universal testing machine (Instron Model 5566, Instron, Buckinghamshire, UK) at a crosshead speed of 1 mm/min. Fracture toughness was calculated from equation (2).

$$K_{IC} = 0.59 \left( \frac{E}{H} \right)^{1/8} \left( \frac{\sigma_f P^{1/3}}{c^{3/4}} \right)$$  \hspace{1cm} (2)$$

where $\sigma_f$ is the stress at failure.

Group III: Fractographic approach —The fracture surfaces of all tested specimens from group II were observed under optical microscopy (Nikon TMS) to determine the critical flaw sizes that originated from Vickers indentations, and the fracture toughness was calculated from equation (3).

$$K_{IC} = \psi \sigma_f c^{1/2}$$  \hspace{1cm} (3)$$

where $\psi$ is a numerical constant that accounts for loading and crack geometry (1.65 for indentation cracks), and $c$ is the radius of an equivalent semicircular or semieliptical crack (equal to $(ab)^{1/2}$), where $a$ is the semi-minor axis of an elliptical crack, and $b$ is the semi-major axis of an elliptical crack (Fig. 1).

Group IV: Surface crack in flexure (SCF) —Indentation cracks were made using a Knoop hardness indenter at loads of 48.5 N and 29.4 N for IPS Empress® Esthetic and IPS e.max® Ceram, respectively. These loads were selected such that the most critical flaw was created without serious damage to the specimens. The load was applied with the long axis of the indenter perpendicular to the long axis of the test specimens. A full-force dwell time of 15 s was used. The indented surfaces of specimens were slightly ground to a depth of 4.5 to 5 times the indentation depth to remove the residual stress fields near the initial flaw that were produced by the indentations. The approximate depth (h) of the Knoop impression can be calculated from the length of the long diagonal (D) as $h = D/30$ (Fig. 2). After indented surface removal, the 10 bar specimens from both materials were subjected to 4-point flexure test, and the fracture toughness was calculated from equation (4).

$$K_{IC} = Y \sigma_f c^{1/2}$$  \hspace{1cm} (4)$$

where $Y$ is stress intensity factor coefficient that is calculated according to ASTM c 1421-01b$^{9}$.

**Statistical analysis**

The differences between the mean fracture toughness values were analyzed using a one-way ANOVA with Dunnett’s test at a significance level ($\alpha$) of 0.05 using SPSS 13 (SPSS, Chicago, IL, USA).

**RESULTS**

Figures 3 and 4 show the optical micrographs of the fracture surfaces of IPS Empress® Esthetic and IPS e.max® Ceram from groups III and IV, respectively. Compared with group III, there is no depression from indentations left in group IV. The critical flaw size was measured and used in the fracture toughness calculation. The fracture toughness is given in Table 2.

For IPS Empress® Esthetic, the fracture toughness values for group II and group III did not differ from that of group IV (standard technique) ($p = 0.15$ and $0.41$, respectively), while the value in group I was significantly
Fig. 3 Illustrations of critical flaw size of specimens in group III, (A) IPS Empress® Esthetic, and (B) IPS e.max® Ceram. Arrows shows the Vickers indentation on the surface.

Fig. 4 Optical micrographs of the fracture surface of the specimens in group IV, showing the critical crack of specimens from (A) IPS Empress® Esthetic, and (B) IPS e.max® Ceram. Arrows illustrate critical crack boundaries, and there is no Knoop indentation after surface grinding.

Table 2 Mean and standard deviation [SD] of fracture toughness of the materials in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Fracture toughness [SD] (MPa·m$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Group I</td>
</tr>
<tr>
<td>IPS Empress® Esthetic</td>
<td>1.24 [0.10]*</td>
</tr>
<tr>
<td>IPS e.max® Ceram</td>
<td>0.90 [0.05]*</td>
</tr>
</tbody>
</table>

* Fracture toughness was different from that resulting for the standard technique (Group IV)

different from that for group IV ($p<0.05$).

For IPS e.max® Ceram, the fracture toughness values of groups I and II were significantly different from that of group IV ($p<0.05$), whereas, for group III, the $K_{IC}$ resulting from the technique using fractography did not differ from that for the standard technique ($p=0.20$).

DISCUSSION

In this study, it was found that the $K_{IC}$ obtained from IF technique was statistically different from that of the standard technique in the Empress® Esthetics group, and the $K_{IC}$ obtained from the IF and IS techniques were also different from that of the standard technique; therefore, the null hypothesis was rejected.

The SCF method is a standard method, which was
used to determine $K_{IC}$ in the control group of this study. The SCF method provides a short precrack to avoid R-curve behavior. However, a critical step is to remove residual stresses around the Knoop indentation, which requires fractographic experience to locate the critical crack boundary, especially for ceramics with porosities or a coarse grain microstructure. The critical crack can be readily seen in materials that contain grain sizes less than 10 $\mu$m. The precrack in coarser grained materials may be discernible, even though 50–150 N-loads are recommended to ensure that a halfpenny crack is created. In this study, preferred loads for Empress® Esthetic and e.max® Ceram were 49 N and 29.4 N, respectively.

In this study, there was no agreement between the $K_{IC}$ values for the IF and SCF techniques, the standard technique and IF methods seem to yield a greater $K_{IC}$ value than that for the SCF method. Although the crack length in our studies was measured 1 h after indentation to ensure that there was complete crack extension, the $K_{IC}$ value was still overestimated comparing with the SCF technique. In a pilot study, we found that the crack measurement immediately after indentation was difficult to obtain because of ongoing crack extension. This may increase more variation about the timing of crack measurement, although intermediate measurement has been suggested by using inert environment such as silicone oil. However, the crack was not always readily discernable. In addition, the crack extension in our pilot study was observed immediately, at 5 min, 10 min, 15 min, 30 min, 1 h, and 24 h after indentation, and we found that crack length at 1 h was comparable to crack length at 24 h after indentation. Therefore, the crack measurements of the two ceramics in this study were made approximately 1 h after indentation. Nevertheless, if the crack length was measured instantaneously after indentation, the $K_{IC}$ would be greater because of the shorter crack length. This would overestimate the $K_{IC}$ of materials in this study unless the residual tensile stress was present.

Fischer and Marx determined the $K_{IC}$ of various dental ceramics using the IF technique compared with the standard single-edge-V-notched beam technique, and they found that a universal constant or prefactor used in equation 1 was material-specific, and the constant needed to be modified according to material and stress condition. They concluded that this technique could only be used as a rough estimation of the $K_{IC}$, and should not be used for unknown ceramic materials.

The IS technique requires bar-shaped specimens with polished surfaces. The specimens are indented at the center of the tensile surface by a Vickers microhardness indenter, then tested using either a 3 or 4-point-bending test. The crack length measurement is not necessary, but the penny-shape crack system is still required. It was found that the crosshead displacement speed can cause statistically different results of fracture toughness obtained with the IS method. Wang et al. studied the effect of testing configurations (3-point, 4-point and biaxial bending tests) and crosshead speeds, and they found that testing configurations had no effect on the fracture toughness; however, the crosshead speeds affected fracture toughness derived from the IS method.

For all the non-standard tests in this study, it was shown that the fractographic approach (group III) yielded a $K_{IC}$ value that was comparable to that of the control group. This method used a similar approach to determine $K_{IC}$, such as a controlled surface flaw that was introduced in the tensile loading site. The same assumption was made that crack is small compared to the thickness of the specimen. However, the geometrical factor ($\psi$) was calculated based on Randall’s interpretation of Irwin’s work, the semicircular crack and residual stress. The value 1.65 was used in calculation. In addition, the procedure that was based on fractographic analysis is simpler than that for the standard method. For the SCF method, the indented and residual stress zone of the specimen must be removed to a depth that is calculated from the long diagonal of the Knoop microhardness indenter. Additionally, the surface parallelism should be maintained during polishing. Therefore, the fractographic approach is not a more complex method to determine the fracture toughness compared with the standard technique.

The fractography techniques presented in our study was based on the fracture mechanics theories, and it was recommended for determining the fracture toughness of the brittle materials such as ceramics, glass and glass-ceramics. In our study, two types of dental ceramics were used for comparison because they were readily discernible in all experimental groups and less complicated compared with the polycrystalline ceramics. Therefore, with the limitation of this study, there should be further studies on a variety of brittle materials such as polycrystalline ceramics.

None of the procedures is an absolutely straightforward method to determine the fracture toughness. The fracture toughness determination is sensitive and different methods employ different processing procedures and test parameters used that affect accuracy. Consequently, test selection should be based on a sound understanding of the theoretical concept and inherent limitations of each technique.

**CONCLUSIONS**

Within the limitations of our study, the following conclusions are drawn.

1. Indentation methods tend to yield a greater $K_{IC}$ value than that of the standard technique used for the materials in this study. They should not be used to determine $K_{IC}$ because of technical limitations. It should only be used for rough screening because of its expediency, simple specimen preparation, and minimal financial cost.

2. The $K_{IC}$ value, which was derived from fractographic approach, was not significantly different from that obtained from the SCF method, the standard technique. This technique may be
employed as a reliable method to investigate the $K_C$ of ceramic materials.

**ACKNOWLEDGMENTS**

This study was supported by a Research Grant MRG5380265 from the Thailand Research Fund and the Department of Prosthodontics, Faculty of Dentistry, Mahidol University.

We also would like to thank the Research Center of Mahidol University, the National Metal and Materials Technology Center (MTEC), Thailand, and the Department of Materials Science, Faculty of Science, Chalalongkorn University, for their technical support and specimen preparation.

**REFERENCES**


