Fracture Toughness and Fractography of Dental Ceramics

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Abstract

Chevron-notch short-rod fracture toughness ($K_{\text{ic}}$) and scanning electron microscopy analyses of leucite-, tetrasilicic fluormica-, and alumina- reinforced dental ceramics and control materials were investigated. Short-rod fracture toughness is a measure of the bulk resistance to crack propagation but not of the surface resistance to crack initiation. Results indicated significant differences in $K_{\text{ic}}$ among the following six groups (from lowest to highest (a) Dicor and Optec, (b) Optec, Excelco’s Incisal, Macor and Excelco’s Gingival, (c) Excelco’s Brush-O-Paque, (d) Vitadur-N core, (e) Corn ing’s 9606 glass-ceramic, and (f) Vita Hi Ceram. Good agreement occurred with published data for Macor and 9606. Comparisons of $K_{\text{ic}}$’s to published bending properties revealed poor correlation with both Dicor and Optec. This was attributed to sample geometry and surface preparational differences between short-rod and bending samples. Fractography analysis revealed the brittle nature of the glassy matrix with all fractured surfaces. The alumina particles inhibited crack propagation by pinning the crack at the particle-matrix interface. The fluormica and leucite phases revealed a higher incidence for cleavage fracture. The opaque particles offered some reinforcement effect.

Introduction

The use of porcelain and glass-ceramics in restorative dentistry is increasing due to (a) improved and new formulations and processing techniques, (b) improvements in mechanical behavior, and to their abilities to (c) mimic the appearance of natural teeth, (d) maintain favorable esthetics, and (e) remain relatively bioinert as compared with many types of metallic and polymeric materials [10, 14, 26]. Perhaps their main shortcoming is their tendency to absorb only low quantities of strain energy prior to brittle fracture at a critical strain of about 0.1 % [10] which is found in improperly organized occlusal schemes and bruxism where cusp and incisal edges easily fracture [25]. This is brought about by the growth of subcritical size flaws to critical dimensions by the interaction of the oral fluids with residual or biting stresses [10]. Incisal and gingival (body) porcelains and glass-ceramics transmit biting forces directly from the contacting areas, while the opaque and aluminous core porcelains, being part of the substructure, transmit them indirectly. The hardness and abrasiveness of many dental ceramics can also generate problems where the opposing dentition is not porcelain [25].

Fracture toughness ($K_{\text{ic}}$) has become acceptable for evaluating the strain energy absorbing capacity of materials [3]. It has been shown that the aluminous porcelains possess significantly higher $K_{\text{ic}}$’s than the feldspathic conventional porcelains [15]. Significant differences occurred among particular feldspathic porcelains [11], as well as among experimental dental glasses of varying compositions [20]. The storage in deionized water and artificial saliva has been shown to lower the $K_{\text{ic}}$’s of aluminous porcelains [13]. Residual compressive stresses in porcelain fused to metal restorations were shown to nearly double the apparent fracture toughness of the surface porcelain bonded to the metal [21].

Fracture toughness of dental ceramics has been mainly evaluated with a microindentation crack length technique. This is particularly important with dental ceramics, since a final glazing firing procedure is usually

Key Words: Fracture toughness, fractography, short-rod, chevron-notch, scanning electron microscopy, dental porcelain, aluminous core porcelain, glass-, castable-, and reinforced-ceramics, leucite, feldspar, alumina, tetrasilicic fluormica.
The purpose of this project was to evaluate the bulk fracture toughness of most currently used types of dental ceramics. These include the feldspathic and aluminous core porcelains as well as a castable glass-ceramic. Fractography analysis by qualitative electron microscopic examination of the fractured surfaces [19] was also conducted to compare surface roughness, mode of crack propagation, and reinforcement effect from the thermally formed or additive phases. Documentation of fractured surfaces generated under plane strain conditions also forms a source of information for use in comparisons to fractured surfaces of dental ceramics retrieved from in-service conditions.

Besides the microindentation fracture toughness technique, at least four additional independent techniques have been used for glass and ceramic materials [3]. These include the double cantilever beam (DCB), double torsional plate, single-edge notch beam (SENB), and chevron-notch beam, bar or rod. Both the DCB and double torsional geometries require precracking prior to testing. The SENB requires an initial notch cut as well as a sharp crack produced at the base of the notch with a length about one half the notch radius. With the chevron-notch design, the distance from the end face to the apex of the chevron plane is the initial crack length. Therefore no precracking is required. The $K_{IC}$'s for both the chevron-notch and double torsional geometries are independent of crack length. Variants of the conventional DCB sample design include the tapered DCB and the constant-moment DCB, both of which produce $K_{IC}$'s independent of crack length. Compact tension (CT) samples with dimensions much smaller than those of the conventional DCB dimensions have also been used.

A sample geometry for $K_{IC}$ evaluation was sought that (a) approximated the mass of porcelain used in a typical crown form, (b) provided minimal preparational difficulties, (c) provided minimal testing and measurement difficulties, and (d) provided a fracture surface area applicable to electron fractography. The DCB, CT and double torsional geometries were eliminated due to size and/or precracking requirements. Both the SENB and the chevron-notch geometries satisfied most of the above requirements. The short-rod chevron-notch method was finally chosen because a small diameter rod sample could be used which approximated the mass of porcelain in a typical crown.

### Materials and Methods

#### Materials

Table 1 identifies the porcelains and glass-ceramics used. All materials except glass-ceramics 9658 (Macor) and 9606 are used in the fabrication of dental components. Macor and 9606 were included in this project to serve as controls for fracture toughness, since their $K_{IC}$'s have been reported [18].

#### Samples

Figure 1 presents a schematic diagram of the short-rod sample geometry used for evaluating fracture toughness. A discussion of the same short-rod sample geometry and testing procedures which were used with dental cements and filling materials can be found in a prior report [16]. The diameter (B), length (W), and distance to the apex of the chevron plane ($a_c$) were $6.35 \pm 0.03$, $9.53 \pm 0.06$ and $3.37 \pm 0.06$ mm respectively. The chevron angle was $55 \pm 1^\circ$ and the thickness of the chevron slots was $0.18$ mm. The square end groove measured $1.97$ mm wide by $1.73$ mm deep.
Core drilled sample lengths of Dicor, Macor and 9606 measuring 6.35 mm in diameter by 25-30 mm in length were provided by the manufacturer. All glass-ceramics were in a cerammed condition normally used with these materials [9]. For Dicor this consisted of 55% crystallinity by heat treatment at 1075°C for 6 hours. For all other ceramics, the materials were supplied in powder form. Several drops of deionized water or the special modeling liquid were added to the powders to form a slurry which was then added to ground stainless steel molds ranging in diameter between 7.10 and 7.35 mm depending on the porcelain being processed. A larger sized pre-firing diameter was required so that the required 6.35 mm diameter was obtained after firing because of thermal contractions. Ground stainless steel plungers were inserted into both ends and placed in a hydraulic press under 100 kg for 1 minute prior to ejection of the condensed samples from the molds. The as-supplied samples from the manufacturers and the fabricated samples from the ceramic powders were first cut-off and/or ground with diamond wheels to the required length followed by light abrasion on No. 600 grit SiC paper. The square end groove and symmetrical chevron slots separated by an angle of 55°C were easily machined into the samples by the use of diamond wheels with the TerraTek Systems model 4901 Fractometer specimen saw. This device supplied the necessary jigs to hold the samples so that the required positioning and angles were obtained. All prepared samples were stored under laboratory conditions of 23 ± 2°C and 55 ± 5% relative humidity between 1-10 weeks until testing commenced.

Fracture Toughness

Plane strain fracture toughness for short-rod chevron-notch samples ($K_{tv}$) was determined with an elastic-plastic analysis for smooth crack growth [1] by using the TerraTek Systems model 2101A ultra low range Fractometer II Machine. The testing methodology, data reduction and calculation procedures used previously with cement and filling materials and used here with the ceramics have been previously described [16]. Briefly, a Frackjack being part of the Fractometer machine applied a force at the rate of $0.5 \times 10^{-3}$ mm/sec to the inside of the square end groove perpendicular to the chevron plane, thus generating a shearing force commencing from the apex along the chevron plane. This responded to mode I loading. As part of the Frackjack, both a load cell and a crack mouth opening gauge monitored the applied load versus the crack mouth opening displacement. Plots of these type usually reveal, as shown in Figures 2-7, (a) an initial region where the applied load is linear with the opening displacement, (b) a region where "pop-in" of the crack at the apex occurs (small reduction in load), (c) a region where stable crack growth occurs (load increases non-linearly with displacement), and by (d) a peak load followed by decreasing loads leading to unstable crack growth and catastrophic fracture. For materials fracturing via smooth crack growth, two unloading-reloading cycles were made at...
approximately $1.2r_c$ ($r_1$) and $0.8r_c$ ($r_2$), where $r_c$ was the critical slope ratio defined [1] as 0.52 for the particular geometry used. All slope ratios corresponded to the fraction of the initial loading slope ratio $r_0$, which equaled 1. For $r_1$ and $r_2$ the lines were drawn from high points on the curves where reversal in the load was started to low points on the reloading line equating one-half the initial reversal height. The intersection of $r_c$ with the load-displacement plot (considered a continuous extension of the curve if $r_c$ intersected a region of the reloading cycle) defined the critical load ($P_c$). For materials fracturing via a crack jumping process, slope ratios for crack jumps with an accompanying reduction in load by at least 5% between $1.2r_c$ and $0.8r_c$ were determined either by direct unloading-reloading cycles or by vertical interpolation.

Slight revisions, however, were applied to the previously used [16] calculation procedures for the short-rod fracture toughness to accommodate the newly adopted American Society for Testing and Materials Standard E-1304-89 [1]. Fracture toughness for smooth crack growth ($K_{IC}$) was calculated from the following equation,

$$K_{IC} = \frac{Y_M^{*}P_cB}{W}$$

where $Y_M^{*}$ was the compliance calibration for the chevron-notch rod geometry used and equaling the minimum stress intensity factor as a function of crack length. Its value was 29.21. The critical load ($P_c$) occurred at a critical crack length $a_c$ where $r_c = 0.52$. Diameter and length corresponded to B and W respectively.

Analysis of variance (ANOVA) and sample mean comparisons of the data were determined with the use of the PC based program of Statistix 3.1 from Analytical Software [2]. The calculated fracture toughness results for each ceramic were first analyzed in conformity to a normal distribution by determining the Wilk-Shapiro statistic. A two-way analysis of variance with $K_{IC}$ as the dependent variable, ceramic type and replication as the main effect variables, and an interaction term between ceramic type and replication followed. Since an unequal sample size resulted among the different ceramic types, the Statistix program supplied the missing values by least square estimates. Multiple comparison of the means was performed with the Tukey test using a rejection level of 0.050.

Fractography

Fractographic analysis was conducted with the fractured surfaces on the sample halves from fracture toughness testing and with polished cross sections of partially fractured samples perpendicular to the chevron plane. The polished cross sections were prepared by taking intact short rod samples that had been partly fractured with pre-peak loads, mounting in resin, cross-sectioning at locations along the length of the samples that would best reveal the cracks, grinding and polishing to a 1 µm diamond finish. The polished cross sectional surfaces were etched with a 1% hydrofluoric acid solution for times varying up to several minutes. All fractographic samples were sputter-coated with a thin film of gold prior to scanning electron microscopy (SEM) analysis which utilized a Cambridge Stereoscan Mark II instrument. Energy dispersive spectroscopy (EDS) was used to qualitatively detect spectral characteristics of the different phases on the fractured surfaces.

Samples for electron fractography were representative of the samples tested for fracture toughness. Each sample was examined by optical microscopy at both low and high power with a Leitz Orthoplan microscope to reveal the appearance of the fractured surface characteristics. Representative surfaces were then selected for SEM study. For most materials, numerous sample halves were eventually analyzed by electron microscopy. This consisted of approximately five different surfaces for Dicor, Optec, Excelco Opaque, Excelco Gingival and Vita Hi Ceram. Similarly, approximately five polished cross sectional samples each for Dicor, Optec, Excelco Opaque, Excelco Incisal and Vita Hi Ceram were analyzed.

Results

Fracture Toughness

Figures 2-7 present typical load-crack mouth opening displacement plots for Excelco Incisal, Optec Body, Dicor, Vita Hi Ceram, Macor and 9606. Plot shapes for Excelco’s Gingival and Opaque were similar to Incisal, and Vitadur-N core similar to Vita Hi Ceram. In all plots, two unloading-reloading cycles with their slope ratios of $r_1$ and $r_2$ are shown, along with the initial slope ratio, $r_0$ and the critical slope ratio $r_c$. For the most part, all ceramics were characterized on the load-displacement plots by mainly an initial linear region, a point where crack 'pop-in' at the apex of the chevron plane occurred, and by stable crack growth over about one-third the length of the plane prior to catastrophic failure. Table 2 presents data pertaining to the load-displacement plots. Included are the number of samples tested ($n_t$), number of samples satisfying the conditions of the test ($n_s$), the mean critical load ($P_c$), the mean maximum load developed in the test ($P_m$), the mean plasticity ($p$), the mean fracture toughness ($K_{IC}$), and the Wilk-Shapiro statistic for fracture toughness ($W-S$).
K\textsubscript{F}\textsubscript{v}-Fractography of Dental Ceramics

![Graphs showing force-displacement curves for different dental ceramics.](image-url)
TABLE 2: Fracture Toughness Data

<table>
<thead>
<tr>
<th>Material</th>
<th>( n_t )</th>
<th>( n_s )</th>
<th>( P_t (N) )</th>
<th>( P_m (N) )</th>
<th>( p )</th>
<th>( K_p (MNm^{-1.5}) )</th>
<th>( W-S^{**} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dicor</td>
<td>14</td>
<td>6</td>
<td>27.4 (1.84)</td>
<td>28.1 (1.36)</td>
<td>0.02 (0.092)</td>
<td>1.31 (0.088)</td>
<td>0.976</td>
</tr>
<tr>
<td>Optec Body</td>
<td>16</td>
<td>13</td>
<td>29.4 (1.36)</td>
<td>29.9 (2.06)</td>
<td>0.05 (0.092)</td>
<td>1.41 (0.065)</td>
<td>0.958</td>
</tr>
<tr>
<td>Incisal</td>
<td>11</td>
<td>5</td>
<td>31.3 (1.64)</td>
<td>31.6 (1.50)</td>
<td>0.02 (0.032)</td>
<td>1.50 (0.078)</td>
<td>0.952</td>
</tr>
<tr>
<td>Excelco Incisal</td>
<td>17</td>
<td>11</td>
<td>33.2 (2.01)</td>
<td>34.7 (2.57)</td>
<td>0.01 (0.067)</td>
<td>1.59 (0.096)</td>
<td>0.914</td>
</tr>
<tr>
<td>Gingival</td>
<td>15</td>
<td>10</td>
<td>33.4 (1.98)</td>
<td>34.8 (2.20)</td>
<td>0.07 (0.098)</td>
<td>1.60 (0.095)</td>
<td>0.919</td>
</tr>
<tr>
<td>Macor(9658)</td>
<td>13</td>
<td>8</td>
<td>33.6 (1.73)</td>
<td>35.0 (1.63)</td>
<td>0.11 (0.077)</td>
<td>1.61 (0.083)</td>
<td>0.925</td>
</tr>
<tr>
<td>Excelco Opaque</td>
<td>17</td>
<td>12</td>
<td>39.2 (2.41)</td>
<td>42.5 (3.55)</td>
<td>0.00 (0.076)</td>
<td>1.88 (0.115)</td>
<td>0.956</td>
</tr>
<tr>
<td>V'dur core</td>
<td>9</td>
<td>6</td>
<td>50.4 (2.34)</td>
<td>52.4 (2.20)</td>
<td>0.02 (0.072)</td>
<td>2.41 (0.112)</td>
<td>0.957</td>
</tr>
<tr>
<td>9606</td>
<td>11</td>
<td>9</td>
<td>54.9 (6.96)</td>
<td>58.7 (4.58)</td>
<td>0.01 (0.119)</td>
<td>2.63 (0.331)</td>
<td>0.965</td>
</tr>
<tr>
<td>V Hi Ceram</td>
<td>11</td>
<td>5</td>
<td>61.1 (1.69)</td>
<td>62.4 (2.35)</td>
<td>0.00 (0.033)</td>
<td>2.92 (0.080)</td>
<td>0.939</td>
</tr>
</tbody>
</table>

* means and standard deviations; a line connecting means denotes no significance at a rejection level of 0.050; the critical value for comparison was 0.223 MNm\(^{-1.5}\)

** Wilk-Shapiro statistic

Standard deviations are presented after each mean. Since the number of samples complying with the requirements of the test varied with the ceramic material being tested, the total number of samples tested per ceramic type varied to insure a sufficient number of valid samples. Results from the Tukey multiple comparison of the means analysis are presented in Table 2 as vertical lines between means. A line connecting means denoted no significance between those means. Six significantly different groups existed with Vita Hi Ceram the highest, which was followed by 9606, Vitadur-N core, Excelco Opaque, a group consisting of Excelco Gingival, Macor, Excelco Incisal, Optec Incisal and Optec Body, and a group consisting of Optec Incisal, Optec Body and Dicor the lowest in that order. The high value for the Wilk-Shapiro statistic with all ceramics indicated the sample data approximated normal distributions so justifying the use of the parametric ANOVA and Tukey multiple comparison of the means test.

Fractography

Figures 8-26 present SEM micrographs for either the fractured surfaces or the polished cross sections perpendicular to the chevron plane. Figures 8-11 reveal low magnifications for the fractured chevron plane surfaces from Excelco's Opaque, Optec, Dicor and Vita Hi Ceram respectively. At this power, Excelco's Gingival and Incisal fractured surfaces appeared similar to the former and Vitadur-N core similar to the latter. Macor and 9606 ceramics appeared at low power with relatively smooth fractured surfaces between those for Optec (Fig 9) and Dicor (Fig 10). Vita Hi Ceram as with Vitadur-N core revealed porosity (labeled P in Fig 11). Figure 12 revealed a low power polished cross section for Optec. As with all valid tests, the crack (labeled C) propagated in a flat manner between the two chevron slots (labeled S). Figures 13-15 present intermediate power micrographs for the fractured surfaces from Excelco's Incisal, Gingival, and Opaque surfaces. All surfaces revealed a smooth glassy matrix phase (labeled G) as well as scattered regions with irregularities (labeled L). Qualitative EDS analysis detected Al, Si and K comprising the irregular surface features, which when taken as their oxides, constitute the chemical ingredients of leucite. Figure 16 revealed the characteristics of the Incisal surface at higher magnification. In addition to smooth features within the glassy matrix, striations (labeled S) and brittle cleavage-like features (labeled B) also occurred. For the Excelco Opaque fractured surface, Figure 17 revealed the presence of opaque particles (labeled P). Figure 18 presents a polished cross section for Excelco's Incisal, which was similar to the appearance for Excelco's Gingival and Opaque surfaces. The crack (labeled C) has propagated mainly around leucite reinforcement phase (labeled L) close to the interface between the leucite and the surrounding glass matrix (labeled G). This has given rise to the irregular regions noted on the fractured surfaces. Figures 19 and 20 present micrographs for the fractured surface and polished cross section from Optec. Feldspathic
porcelains, such as Optec revealed both a smooth glassy matrix (labeled G) and regions with irregularities (labeled L) which were again, in conjunction with qualitative EDS, reasoned to be leucite. These latter regions, however, appeared in a more orderly fashion

Figures 8-11. Low magnification micrographs of fractured sample halves of: Excelco's Brush-O-Paque (Figure 8); Optec (Figure 9); Dicor (Figure 10); and Vita Hi Cream (Figure 11).

Figure 12. Low magnification micrograph of polished section perpendicular to the chevron plane for an intact fractured Optec sample. The crack front (labeled C) has propagated between the two chevron slots (labeled S). A polishing scratch extends from the right slot downward and to the left.

with Optec, as depicted by the region labeled L in Figure 19. Figures 21 and 22 present micrographs for the fractured surface and polished cross section from Dicor. The prismatic flat "plate-like" nature of the reinforcement crystallites were confirmed by qualitative EDS analysis to contain Si and F, which were reasoned to be the tetrasilicic fluormica crystallites (labeled M). The polished cross section revealed the crack front (labeled C) had propagated through both glassy matrix
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Figures 23-26 present micrographs for the fractured surfaces and polished cross sections from Vitadur-N core and Vita Hi Ceram respectively. Figures 23 and 25 revealed the brittle nature of the glassy matrix fracture (labeled G) and the appearance of the alumina phase (labeled A) for both ceramics. The latter phase was analyzed by EDS to contain Al, which when taken as its oxide, corresponded to the alumina phase. Figures 24 (labeled G) and to some extent fluormica crystals (labeled M).

Figures 23-26 present micrographs for the fractured surfaces and polished cross sections from Vitadur-N core and Vita Hi Ceram respectively. Figures 23 and 25 revealed the brittle nature of the glassy matrix fracture (labeled G) and the appearance of the alumina phase (labeled A) for both ceramics. The latter phase was analyzed by EDS to contain Al, which when taken as its oxide, corresponded to the alumina phase. Figures 24
Figure 19. Micrograph of an Optec fractured surface revealing glassy matrix phase (labeled G) and leucite phase (labeled L).

Figure 20. Micrograph of an intact fractured Optec polished section perpendicular to the chevron plane revealing glassy matrix phase (labeled G), leucite phase (labeled L), and crack front (labeled C).

Figure 21. Micrograph of a Dicor fractured surface revealing fluororita crystallite plates (labeled M).

Figure 22. Micrograph of an intact fractured Dicor polished section perpendicular to the chevron plane revealing glassy matrix phase (labeled G), tetrasilicic fluororita second phase particles (labeled M), and crack front (labeled C).

Figure 13. Micrograph of Excelco's Incisal fractured surface revealing glassy matrix phase (labeled G) and leucite phase or interface between leucite phase with matrix (labeled L).

Figure 14. Micrograph of Excelco's Gingival fractured surface revealing glassy matrix phase (labeled G) and leucite phase or interface between leucite phase with matrix (labeled L).

Figure 15. Micrograph of Excelco's Brush-O-Paque fractured surface revealing glassy matrix phase (labeled G) and leucite phase or interface between leucite phase with matrix (labeled L).

Figure 16. Micrograph of Excelco's Incisal fractured surface revealing striations (labeled S) and brittle fracture features (labeled B).

Figure 17. Micrograph of Excelco's Brush-O-Paque fractured surface revealing opaques particles (labeled P).

Figure 18. Micrograph of an intact fractured Excelco's Incisal polished section perpendicular to chevron plane revealing glassy matrix phase (labeled G), leucite phase (labeled L), and crack front (labeled C).
Figure 23. Micrograph of a Vitadur-N core fractured surface revealing glassy matrix (labeled G) and alumina particles (labeled A).

Figure 24. Micrograph of an intact fractured Vitadur-N core polished section perpendicular to the chevron plane revealing glassy matrix phase (labeled G), alumina particles (labeled A), and crack front (labeled C).

Figure 25. Micrograph of a Vita Hi Ceram fractured surface revealing glassy matrix (labeled G) and alumina particles (labeled A).

Figure 26. Micrograph of an intact fractured Vita Hi Ceram polished section perpendicular to chevron plane revealing glassy matrix (labeled G), alumina particles (labeled A), and crack front (labeled C).

and 26 revealed the highly selective propagation path through the glassy matrix for both ceramics. Fractography analysis for 9658 and 9606 were similar to the micrographs reported upon earlier [18]. The fractured surface for 9658 revealed extensive cleavage-type fractures due to the mica flakes and similar to that presented here for Dicor. Small elongated second phases were detected with 9606.

Discussion

Load-Displacement Behavior

The load-displacement plots for Vita Hi Ceram, 9606, Vitadur-N core, Macor and Dicor revealed mostly smooth crack growth. Some evidence for crack jumping occurred with Excelco and Optec samples. Since in most cases, the reduction in load due to crack jumping was less than 5% of the maximum applied load, most
of the load-displacement plots were analyzed by the smooth crack growth procedure.

**Plasticity**

Except for Macor, the mean plasticities ranged between 0.00 and 0.07. These low values would be expected for brittle materials such as dental porcelains. A mean plasticity of 0.11 for Macor is likewise expected, since this is an industrial machineable glass-ceramic. Dicor revealed a much lower mean plasticity of only 0.02.

The standard deviations for the plasticities were high, ranging between 0.03 and 0.12. Since the minimal plasticity which can be determined with precision from the load-displacement plots is of the order of 0.01, the relatively large deviations among samples were due to differences among the samples and not to uncertainties in graphical measurements. Figure 27 presents three alternative stress states occurring with short-rod samples. Without residual stresses the normal sample shape occurs (Fig 27b), while with residual stresses the sample will be either in closure or extension (Fig 27a & c). Residual stresses act upon the sample during routine fracture toughness testing especially during the unloading-reloading cycles to alter the load-mouth opening displacement plots as revealed in Figure 28 [4].

**Bending Properties versus Fracture Toughness**

The bending strength for Vitadur-N core being 80-89 % of that for Vita Hi Ceram [23-24] is in line with their fracture toughness results from this project, the former being 83 % of the latter. The modules of rupture (MOR) for an unspecified Excelco feldspathic porcelain being 40 % of that for Vita Hi Ceram [23], is also in line with the 54-64 % range of \( K_{1v} \) for three Excelco porcelains obtained from this project. For Optec, the MOR is reported to be in the 75-86 % range of that for Vita Hi Ceram [23], while a range of only 48-52 % in \( K_{1v} \) was obtained here. For Dicor the MOR varies more depending upon source. Some reports indicated the MOR to be of the same order as the aluminous core porcelains [14, 9], while other reports indicated Dicor to be only 64-90 % [7, 23] of that for Vita Hi Ceram, or to possess a much lower MOR [12], in fact only 37 % of its value as reported above in reference nine. Results from this project indicated Dicor with a \( K_{1v} \) of 45 % of that for Vita Hi Ceram.

Fracturing of short-rod samples occurred in the Fractometer machine with the application of force in an outward direction and perpendicular to the chevron plane. This state of stress was not much different than that developed in 3-point flexural tests used in the preceding reports. Both generated tensile stresses at the point of crack propagation either in the bottom surface layers of bending samples or at the apex of the chevron plane with short-rod samples. In a bend specimen, a}

\[ K_{1v} \]-Fractography of Dental Ceramics

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**Figure 27. Schematic short-rod sample geometry revealing normal condition without residual stress (b), and extension and closure conditions with residual stress (a and c).**

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**Figure 28. Schematic load versus crack mouth opening displacement plots for the conditions presented in Figure 27.**

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...crack is first made to propagate from existing subcritical size surface flaws, while in a short-rod sample crack propagation is more easily induced to occur by the nature of the geometry. In a short-rod sample, the distance from the end face to the apex of the chevron plane can be thought of as the initial crack length. Hence crack initiation does not have to occur but only crack pop-in and crack propagation. Also, the bend samples were provided with a final surface glazing treatment, while the chevron-notch slots of the short-rod samples were ground by diamond wheel without final glazing treatment. It has been shown that the effect of surface condition [10] and the specific technique used for glazing [6] can significantly increase MOR for dental porcelain. When care was taken to ensure that the porcelain surface was optimized, higher strengths were obtained [17]. The fracture toughness test is a measure of the inherent ability for a material to resist crack propagation.

**Fracture Toughness Comparisons**

Good agreement occurred between the short-rod fracture toughness data obtained here to previously reported data [18] for the two control glass-ceramic materials. For Macor and 9606 the current mean results of 1.61 (0.083) and 2.63 (0.331) MNm\(^{-1.5}\) were 1.10 times larger than obtained with short-bar samples. Slightly better agreement (1.01-1.07 times) occurred when compared to results from chevron-notch bend samples.
Comparisons to previously reported fracture toughness for dental ceramics revealed differences. For Vitadur-N core material, 1.48 (0.29) and 1.75 (0.27) MNm$^{-1.5}$ were obtained [15, 13] from micro-indentation and single edge notch sample, respectively, compared to 2.41 (0.112) MNm$^{-1.5}$ from this project. For Vitadur-N and Dicor crowns, apparent fracture toughness from micro-indentation of 1.96 and 2.10 MNm$^{-1.5}$ were obtained [22]. For the Dicor system, this was a 1.6 times increase over what was obtained here. For Excelco’s Gingival, a value of 0.94 (0.20) MNm$^{-1.5}$ by micro-indentation was obtained [15] as compared to 1.60 (0.095) from this project. Better agreement emerged among incisal, gingival, and opaque porcelain values obtained by micro-indentation that took into account the actual measured hardness to modules of elasticity ratios for each sample required in the calculation for fracture toughness [11]. For Will Ceram Incisal, Will Ceram Body (Gingival), and Vita and Will Ceram Opaque porcelains, $K_{IC}$’s of 1.287 (0.056), 1.376 (0.077), 1.684 (0.058) and 1.750 (0.050) MNm$^{-1.5}$ were obtained, respectively. The high fracture toughness value by micro-indentation reported for Dicor prepared crown forms relative to the value reported here was due to the effects of surface glazing and retention of residual stresses within the surface layers. The micro-indentation measurements of $K_{IC}$ for glazed Dicor were actually measuring $K_{IC}$ for a low fusing glass and not Dicor bulk material. Compressive stresses in the outer surface layers were expected due to the multi layering and multi firing procedures related to the preparation of crown forms. This was the reason an “apparent” fracture toughness has been reported.

**Fractography**

**Glassy matrix.** SEM analysis of the fractured surfaces and of the polished cross sections have demonstrated the brittle nature of the fracturing process through the glass matrix of the ceramics, whether leucite, alumina, or fluorormica particle reinforced. Smooth surfaces, striation lines, and ‘cleavage-like’ features (glassy matrix is amorphous [8]) occurred. The striation lines occurred mainly with the feldspathic porcelains and likely resulted from fracturing processes related to crack jumping, since the load-displacement plots for these porcelains revealed a higher incidence of crack jumps.

**Alumina reinforced porcelains.** Distinctions in the mechanism of fracture between Vita Hi Ceram and Vitadur-N core were not detected from fractography analysis. Both resisted crack propagation by pinning the crack at the alumina particles. Because Vita Hi Ceram revealed a higher $K_{IC}$, it was better able to inhibit crack propagation. This was due to differences between the two materials in either the glassy matrix or reinforce-ment phase. Borosilicate and feldspathic glasses containing dissolved alumina wet alumina particles better than glasses not containing dissolved alumina. Improved wetting of the alumina crystals ensures stronger bonding and higher post-sintering densities [14], thus increasing resistance to particle pull-out and inhibiting crack propagation. The type and amount of the fluxes comprising the glass composition are factors controlling firing temperature and viscosity of the melt, which in turn would also be expected to affect interactions with second phase particles. Differences in processing the alumina particles, such as between fused and calcined alumina particles, in surface treatment and modification, and in particle size distributions would also be expected to alter interaction with glassy matrix.

**Tetrasilicic fluormica reinforced porcelains.** The tetrasilicic fluormica crystallites in Dicor in contrast to the alumina particles in Vita Hi Ceram and Vitadur-N core appeared to have a higher incidence of fracture as qualitatively assessed from the electron micrographs. The comparisons between Figure 22 with Figures 25 and 26 were typical of many additional regions analyzed along the crack path. Figure 22 revealed a mica platelet, labeled M, that was cleaved into two pieces by the process of crack propagation, while the alumina particles, as detected from the electron fractographs, were able to better resist fracture. This distinction between materials was reasoned to be the primary factor for the reduced fracture toughness of Dicor relative to the alumina glass composites.

**Leucite reinforced porcelain.** For the feldspathic porcelains, crack propagation occurred around the leucite reinforcement particles to a large extent and also to some degree partly through the particles. Cracks not associated with the actual fracturing process were also detected in some regions, particularly around leucite particles. These were similar in appearance to cracks in micrographs reported earlier [5]. This was related to the effects from the mismatch in thermal expansion coefficients between leucite and the glass phase and due to the displacive martensitic transition of high to low leucite occurring within the temperature range of 400-500°C. For Optec, crack propagation similarly occurred around some of the leucite particles and also partly and directly through other leucite particles. The leucite phase in Optec may be added directly to the powder frit, a possible reason for leucite phase contained in orderly shaped regions. Excelco’s Opaque material revealed the presence of opaquing particles. Both a larger size of the order of 4-6 µm in diameter and a much smaller size grouped in clusters with other particles of the same size were detected. Opaquing particles in dental porcelains include titanium oxide, zirconium oxide, stannic oxide,
and aluminum oxide [26]. The possibility existed that the smaller size particles were colorants, but because of their absence in the Incisal and Gingival forms, they were assumed to be opaquing particles. The higher fracture toughness of Opaque relative to Incisal may have been due to the reinforcement effect from the opaquing particles. Cleavage fracture of the larger-sized opaquing particles was seen.

Conclusions

a. Significant differences in the fracture toughness of dental ceramics occurred, ranging from a low of 1.31 MNm\(^{-1.5}\) for Dicor to a high of 2.92 for Vita Hi Ceram. Feldspathic porcelains ranged between 1.41 to 1.88 MNm\(^{-1.5}\).

b. A comparison of the ranking from the fracture toughness results to the ranking from published modules of rupture data indicated differences for Dicor and Optec. Differences in sample geometry and surface preparation between short-rod and 3-point bending samples probably contributed to these results.

c. Fractographic analysis revealed the glassy matrix to be susceptible to brittle fracture. Reinforcement was brought about by alumina, tetrasilicic fluormica and leucite second phase particles. Opaquing particles in the Opaque porcelain also appeared to provide reinforcement.

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References


Discussion with Reviewers

W. A. Brantely: The equation to determine $K_{LCS}$ from the experimental data should be explained further. Is the value of the compliance constant $A$ provided by Terra Tek or should this constant be established by a calibration procedure? As the author notes, materials 9658 and 9606 can serve as controls since fracture toughness data have been previously reported in Ref. 18.

Author: The procedure used in previous work by this author [16] for calculating $K_{LCS}$ of short rod samples was according to Barker. That is,

$$K_{LCS} = (A) (P_c) (1+p) / B^{1.5}$$

where $A$ is a compliance constant determined experimentally by Barker by using materials of known fracture toughness. For the short rod sample geometry as used here, $A$ equals 22.0. Also paralleling Barker’s work was the work of Shannon, Busbey, and others. They related a dimensionless stress intensity factor ($Y$) to crack length. For most fracture test samples, $Y$ increases continuously with increasing crack length. For chevron-notch specimens, however, the corresponding factor $Y^*$ reaches a minimum $Y^*_m$ at a crack length of $a_m$ as the crack extends along the wedge shape of the chevron plane. The values of $Y^*_m$ and $a_m$ are functions of specimen dimensions and notch geometry only and are independent of materials properties. These relationships have been fitted with polynomial expressions and have been included in the ASTM standard [1].

S. F. Rosenstiel: The seven dental products tested represent different classes of dental ceramics, which have different optical properties and clinical applications. Of the seven, only Vitadur-N core ceramic and Vita Hi Ceram can be said to have essentially comparable usage; the other dental ceramics are quite different. Could the author emphasize the clinical applications and optical characteristics of the different ceramics more fully?

Author: Excelco’s porcelains are intended to be fused as veneers to metal substrates. The opaque porcelain is first applied directly over the metal in an attempt to mask the colors of the metal. As shown in the micrographs, the opaque porcelain contains metal oxide additives. The gingival and incisal porcelains, available in various shade designations, are used to build up the body and surface details of a restoration as life-like as possible. The Optec porcelain is intended for fabricating all ceramic crowns, inlays, onlays, and anterior bridges. Various shade designations are available to build-up life-like details. Shading is accomplished externally. Vita Hi Ceram and Vitadur N though higher in strength and fracture toughness do not transmit and reflect light as the other porcelains (except opaque). These materials are confined to core applications.

Reviewer III: It is implied that leucite is added in a controlled manner directly to the powder frit only for Optec and not for Excelco’s porcelain. What proof is available to support this contention?

Author: Leucite-containing dental porcelains can be produced either by a) the nucleation and growth of leucite phase in a glassy matrix from melting and cooling feldspar prior to frit formation, or b) by the admixing of leucite particles to an already formed glass frit. With both methods, the firing process used will control any additional crystallization of leucite that may occur. Excelco’s porcelain could be produced by either method.

B. K. Moore: In the fractography results and discussion, numerous references are made to the identity of various phases present in the different ceramics. In some cases the question arises how the author knows what these actually are?

Author: Energy dispersive analysis was used in a limited way for qualitative analysis. The fluormica crystallites in Dicor, the alumina particles in the core materials, and the leucite phase in the feldspathic porcelains were identified.