Stress distribution and failure mode of dental ceramic structures under Hertzian indentation

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Abstract

Objectives. To understand better the clinically-relevant failure of the ceramic in ceramic–cement–substrate structures under Hertzian indentation, including the effects of supporting substrate modulus and ceramic thickness on the stress distribution in the ceramic.

Methods. Discs (thickness, \( T_c = 0.2, 0.6, 1.2, 1.6, 2.0, 2.4 \) mm) of a glass–ceramic material (IPS Empress 2, Ivoclar) were cemented (Variolink II, Vivadent) to flat polymer substrates with modulus of elasticity \( E_s \) of 2, 6 and 10 GPa. The top surface of the ceramic–cement–substrate structure was loaded by a 20 mm radius spherical indenter until the initial failure of the ceramic occurred. The finite element method was used to analyse the stress distribution under such Hertzian indentation, varying \( E_s \) and \( T_c \), as well as calculating the maximum tensile stress based on the experimentally observed failure load and contact radius. The failure initiation site of the ceramic was identified by fractography using scanning electron microscopy.

Results. The tensile stress concentration at the cementation surface of the ceramic was the predominant factor controlling the ceramic failure. Failure load increased with increase of \( E_s \), while the maximum tensile stress at the cementation surface of the ceramic clearly decreased. Failure load increased logarithmically with ceramic thickness, but the critical tensile stress increased linearly.

Significance. The failure mode observed clinically for ceramic restorations was reproduced in laboratory tests.

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Keywords: Dental materials; Ceramic strength; Stress distribution; Hertzian indentation

1. Introduction

Ceramic restorations have become popular due to their resemblance to real tooth tissue and are especially used for anterior teeth, but high failure rates have been reported, in particular for crowns on molar teeth. To evaluate the flexural strength of dental ceramics, traditional load-to-failure tests on simply supported beams or discs have been used. However, many important characteristics of the results of such laboratory tests have been inconsistent with those of clinical observation, such as the magnitude of the failure load, initial failure site and the failure mechanism [1–8]. The initiation of failure in such tests has been from indentation damage at the contact surface (i.e. the occlusal surface equivalent) [5,6], although failure has been observed clinically to be initiated at the cementation surface [1,4,9]. Indeed, the finite element method (FEM) has been used to show that tensile stresses are concentrated at the cementation surface of crowns [10,11]. In addition, extremely high loads (1500–5000 N) have been required to cause failure, resulting in many fragments [2,6], while the typical clinical failure yields only two [1,4,7], implying that more energy is stored during such laboratory testing for release at failure than in service, although clinical failure could also be due to slow crack growth.

Recently, Hertzian indentation has been used to evaluate the mechanical properties of dental ceramics [12–21]. It appears to be a useful protocol for the evaluation of behavior related to microstructure in dental materials [22]. The method has been used by applying loads to clinically realistic specimens (e.g. ceramic crowns) by a stiff indenter or a pin having a curved edge [2,3,5,6,23], or by compressive loading against flat plates (e.g. ceramic discs) by a flat-ended pin [15,24]. The fixed contact area of the latter approach means that it is easy to calculate the nominal
stress using FEM, but there is necessarily a stress singularity at the edge of the contact area in such experiments and this tends to cause premature and non-relevant failure.

The purpose of the present investigation was to understand the failure mode of ceramic structures under Hertzian indentation as well as the failure load and the tensile stress for crack initiation at the cementation surface as a function of the substrate modulus of elasticity and ceramic thickness. This was with a view to assisting in improvement of the design of ceramic restorations clinically and increasing their fracture resistance.

2. Materials and methods

2.1. Mechanical tests

Glass–ceramic (IPS Empress 2, Batch No. A23747, Ivoclar, Schaan, Liechtenstein) discs 10 mm in diameter and with thicknesses \( t \) ranging from 0.2 to 2.4 mm were fabricated using the lost-wax process. All procedures followed the manufacturer’s instructions. Patterns were prepared by overfilling holes in sheet poly(methyl methacrylate) of the appropriate thickness, laid on a glass slab, with molten wax (Kerr Inlay wax, Sybron Kerr, Emeryville, CA, USA) after first brushing surfaces with separating agent (Microfilm, KerrLab, Orange, CA, USA). The top surface was trimmed flat when the wax was cold before removing the pattern from the mold. The wax pattern, with a sprue attached at the rim, was invested (Special Investment System, Ivoclar), the investment being mixed under vacuum with its special liquid and poured into a casting ring with deionized water. The ceramic discs were then carefully divested by grit blasting with sand (English Abrasives, London, England), to the required thickness, care being taken to preserve the parallelism of the top and bottom surfaces. To roughen the substrate for bonding with the ceramic, one surface was ground dry on 150-grit abrasive paper (Eagle Brand, Komatsubar). The bonding surface of the ceramic disc was treated with the etching gel for 20 s, rinsed with deionized water and blown dry. The etched surface of the ceramic and the roughened surface of the substrate were silanized (Monobond-S), using a brush to form a thin film, for 60 s, and then dried with water- and oil-free air. Contamination was carefully avoided. The silanized surfaces were then wetted with a film of adhesive (Heliobond) using a brush, ensuring that the film was continuous.

The two parts of the luting cement were mixed in a 1:1 ratio on a mixing pad for 10 s with a spatula. The mixed cement was then loaded with a 10 kg deadweight for 15 min. Finally, a visible-light curing unit (Hilux 200, Express, Istanbul, Turkey) was used for 40 s to polymerize the luting cement uniformly on the substrate and the ceramic placed onto it, the whole structure was then loaded with a 10 kg deadweight for 15 min. The bond strength values were determined using a universal testing machine (Zwick, Ulm, Germany).

The glass–ceramic was bonded to the substrate using the recommended dual-cure filled resin adhesive system (Variolink II, Vivadent, Schaan, Liechtenstein) (Table 2). The procedures followed the manufacturer’s instructions [25]. Ceramic and substrate were cleaned in deionized water for 10 min in an ultrasonic bath (Decon FS 200, Hove, Sussex, England). The bonding surface of the ceramic disc was treated with the etching gel for 20 s, rinsed with deionized water and blown dry. The etched surface of the ceramic and the roughened surface of the substrate were silanized (Monobond-S), using a brush to form a thin film, for 60 s, and then dried with water- and oil-free air. Contamination was carefully avoided. The silanized surfaces were then wetted with a film of adhesive (Heliobond) using a brush, ensuring that the film was continuous.

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Table 1

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Rod diameter (mm)</th>
<th>( E_s ) (GPa)</th>
<th>Cat./batch no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyacrylonitrile–butadiene–styrene (ABS)</td>
<td>10</td>
<td>2</td>
<td>AB 307940/1</td>
</tr>
<tr>
<td>Polycarbonate–20% glass fiber</td>
<td>25</td>
<td>6</td>
<td>CT 327920/1</td>
</tr>
<tr>
<td>Nylon 6.6–30% glass fiber</td>
<td>10</td>
<td>10</td>
<td>AM 367910/2</td>
</tr>
</tbody>
</table>

Table 2

<table>
<thead>
<tr>
<th>Variolink II component</th>
<th>Batch no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>B23328</td>
</tr>
<tr>
<td>Catalyst (low viscosity)</td>
<td>A23650</td>
</tr>
<tr>
<td>Etching gel</td>
<td>B46605</td>
</tr>
<tr>
<td>Monobond-S</td>
<td>C12012</td>
</tr>
<tr>
<td>Heliobond</td>
<td>B44146</td>
</tr>
</tbody>
</table>

was trimmed off when the cement was cured. After bonding the ceramic to the substrate, the margin of the interface around the specimen was sealed (Fuji Varnish, GC Corporation, Tokyo, Japan) to reduce water loss from the luting cement and minimize possible deleterious effects. Bonded specimens were allowed to stand in the air at room temperature (23 °C) for 24 h for the curing reactions to approach completion. Using a screw micrometer, the cement thickness was determined to range from 37 to 62 μm (mean: 50.7 μm).

Sixty ceramic discs 1.0 mm thick were prepared and divided randomly into three groups of 20 for tests of the effect of the supporting substrate modulus of elasticity \( E_s = 2, 6, 10 \text{ GPa} \). For tests of the effect of thickness, specimens were in six groups of 20 at each of \( T_c = 0.2, 0.6, 1.2, 1.6, 2.0, 2.4 \text{ mm} \); \( E_s \) was set at 10 GPa. Data from the 1.0 mm group above with \( E_s = 10 \text{ GPa} \) were also treated as part of this sequence.

The specimen was placed in a steel holder with a flat ground base attached to the moving crosshead of the testing machine (Model 1185, Instron, High Wycombe, England), the center point of the specimen being aligned on the loading axis (Fig. 1). The high-carbon steel spherical indenter, 40 mm diameter, was replaced after 40 specimens with \( T_c \approx 1.6 \text{ mm} \), or 60 thinner specimens, to reduce the effects of wear. Other test conditions were: crosshead speed: 0.1 mm/min; load cell: 5 kN full scale, load range 20 or 50%.

Ceramic failure was observed as a sharp decrease in the load-deflection graph. However, as it is crucial to detect the failure occurring in the ceramic as distinct from other components of the structure, two additional means were used to confirm this. A differential microphone sound recording system [26] was used to pick up the cracking sound when the ceramic failure occurred; this was effective for the thicker ceramic discs (i.e. \( T_c = 1.0–2.4 \text{ mm} \)). For thinner specimens (i.e. \( T_c = 0.2–0.6 \text{ mm} \)) the crack was confirmed to have occurred in the ceramic by direct observation under strong illumination; when a sharp decrease had occurred in the chart recording of load, the disc was unloaded and examined microscopically to check that the crack had in fact occurred in the ceramic.

To measure the contact radius, a slip of dental ‘bite-detection’ paper (AccuFilm I, Parkell, Farmingdale, NY, USA), 10 μm thick, was placed between the indenter and the specimen. The red wax contact markings were observed on the ceramic after loading. The maximum diameter of the circle of the contact marking was measured using a linear vernier microscope to 0.01 mm (Griffin, Loughborough, England).

After testing to failure, five ceramic specimens, still bonded to their substrate, were randomly selected from each group and were placed in a muffle furnace at 220 °C for 5 min to soften the cement so that the ceramic was easily separated from the substrate without any further damage. The ceramic fragments were cleaned in 95% ethanol in an ultrasonic bath for 15 min, repeated once with fresh ethanol, then with deionized water and dried in air. Specimens were prepared for scanning electron microscopy (SEM) by sputtering with gold. Each fragment was fixed on a metal stub with sticky wax, and conductive carbon paint was applied at the edge (Dotite Paint, Carbon-20C, Jeol Datum, Tokyo, Japan). Fracture surfaces were observed under SEM (XL30CP, Philips Electron Optics, Eindhoven, The Netherlands) at ×20 to ×50 magnification.

Rank order fracture data were plotted on a normal probability scale as ordinate (SigmaPlot v6, SPSS, Chicago, IL, USA) after ‘continuity’ correction of the rank: \( y = (x - 0.5) \times 100/n \), where \( n \) is the total number of specimens, \( y \) the cumulative probability and \( x \) the rank of the failure load, \( F_c \). This approach was used in preference to Weibull plots as it involved no assumptions concerning the underlying probability model, less distortion at low cumulative probability, and easier visual assessment of behavior.

### 2.2. FEM modeling

An axisymmetrical model was created based on the dimensions and the structure of the experimental specimens with the cement film thickness set at 50 μm and meshed with triangular elements (Fig. 2). The computer code (FACILE, version 3.0) was created by the Civil Engineering Department, The University of Hong Kong. The validity of the FEM model was checked by comparison of the calculated and measured values of the contact radius \( R_c \). The critical tensile stress \( (S_c) \) was defined as the maximum tensile stress calculated by the FEM with the experimental values of \( F_c \) and \( R_c \) inserted. All other values for the FEM calculation are listed in Table 3. To simplify the FEM...
computation, some assumptions were made: that the interfaces were bonded perfectly (no delamination); that there was frictionless contact between the ceramic and the indenter; that the indenter was rigid enough; and that all materials were isotropic, linear and homogeneous. With respect to the boundary conditions, points on the central axis (i.e. y-axis) of the spherical indenter, ceramic and substrate were fixed horizontally (i.e. in the x-direction). The fineness of the meshing had been subjected to a convergence test to ensure sufficient accuracy.

3. Results

3.1. Mechanical tests

There was reasonable agreement between the calculated and observed values for dependency of contact radius, \(R_c\), on ceramic thickness, \(T_c\) (Fig. 3), although the discrepancy, in the sense of being slightly overestimated by the FEM calculation, increased to about 10% for \(T_c > 2.0\) mm. For the effect of \(E_s\) (Fig. 4), the agreement was similar. The plots of failure probability vs. \(\log(F_c)\) were almost parallel for the three values of \(E_s\) (i.e. near constant ratio s.d./mean), although for \(E_s = 2\) and 6 GPa the lines were nearly superimposed (Fig. 5(b)). These plots for the various values of \(T_c\) are shown in Fig. 6(b); again, near constant s.d./mean was found. \(\log(F_c)\) appears to depend linearly on \(T_c\), at least in the range examined \((r^2 = 0.958, P < 10^{-13})\) (Fig. 6(a)).

3.2. FEM modeling

The calculated tensile stress distribution is shown for the three experimental substrate moduli at a load of 500 N in Fig. 7. From data such as this was extracted the maximum critical tensile stress, \(S_c\), which was found to lie on the load axis at the cementation surface, which is therefore where the initial crack of the ceramic was expected to occur. The tensile stress contours did not extend to the contact (top) surface, and although a small tensile region existed outside the contact, circle (not visible) the maximum value never became critical.

The influence of \(E_s\) on \(S_c\) is shown in Fig. 5(d); these plots were divergent even on the log scale, i.e. larger scatter at low modulus. The plots of failure probability against \(S_c\) (Fig. 6(d)) are close to parallel for a linear abscissa, that is to say that the scatter seems independent of ceramic thickness. These calculated values of \(S_c\) are linearly dependent on \(T_c\) \((r^2 = 0.932, P < 10^{-13})\) (Fig. 6(c)).

3.3. Fractography

The SEM observations (Fig. 8) indicated that failure was initiated from the cementation surface of the ceramic for all values of \(T_c\) and \(E_s\).

4. Discussion

The Hertzian indentation mode of testing proved to be straightforward to implement. Unambiguous results were obtained, with the mode of failure resembling that observed clinically. The results suggest strongly that it is tensile stress that is the predominant factor controlling the initial failure of ceramic structures, and the value of \(S_c\) is dependent on the mismatch between \(E_s\) and \(E_c\) (ceramic modulus). In view of this, given the attempted mimicry of oral loading conditions, it appears to be the method of choice for such studies, including access to all the major variables of actual service.

The experimental failure load \((F_c)\) was clearly dependent on ceramic thickness \((T_c)\) (Fig. 6(b)) and, furthermore, failure of the ceramic was initiated at the cementation surface. This is consistent with the findings by Tsai et al. [15] and Chai et al. [27], except for \(T_c = 2.4\) mm where it was found that the initial crack in the ceramic was at the contact (top) surface, rather than at the cementation (bottom) surface. This discrepancy may have arisen from the use of a different radius of indenter: it has already been reported that the failure load for contact surface failure is...
dependent on the radius of the indenter and independent of \( T_c \) \[28\]. In the present study, in order to ensure that \( R_c \) was in the range of the ‘wear facets’ of clinical observation, and to avoid contact surface failure, an indenter of radius 20 mm radius was used, while Chai et al. used a 3.96 mm radius indenter. Harvey et al. \[12\] have said that contact radius has often not been controlled in ordinary load-to-failure laboratory tests, and that it has actually been ignored as a variable in some comparisons of results between studies. From the viewpoint of clinical dentistry, this variable

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**Fig. 3.** Contact radius vs. failure load for various ceramic thicknesses \((T_c)(E_s = 10 \text{ GPa})\). Results for other thicknesses similar. Open symbols and fitted lines: FEM results. Solid symbols: experimental results.

**Fig. 4.** Contact radius vs. failure load for tested substrate moduli \((E_s)(T_c = 1.0 \text{ mm})\). Open symbols and fitted lines: FEM results. Solid symbols: experimental results.
Fig. 5. Experimental test results: effect of substrate modulus, $E_s$. (a) Mean failure load, $F_c$ (error bars: ±1 s.d.). Predicted form of the relationship from Ref. [18], scaled to match experimental results. (b) Failure probability vs. $F_c$. (c) Mean calculated critical tensile stress, $S_c$ (error bars: ±1 s.d.) for observed contact radius. (d) Failure probability vs. $S_c$.

Fig. 6. Experimental test results: effect of ceramic thickness, $T_c$. (a) Mean failure load, $F_c$ (error bars: ±1 s.d.). Predicted form of the relationship from Ref. [18], adjusted to fit three greatest thickness results. (b) Failure probability vs. $F_c$. (c) Mean calculated critical tensile stress, $S_c$ (error bars: ±1 s.d.) for observed contact radius. (d) Failure probability vs. $S_c$. 

appears to be a crucial influence on stress distribution and further investigation seems warranted. $S_c$ was found to depend linearly on $T_c$ (Fig. 6(c)). This is in contrast with the results of Tsai et al. [15] who found that $S_c$ became almost constant for $T_c > 1.6$ mm. This difference might be attributed to their use of a flat-ended pin, but the possibility of a transition at some sufficiently great $T_c$, depending on $R_c$, needs to be considered.

Recently, Rhee et al. [18] have reported, on physical grounds, the following relationship (in our notation)

$$F_c = \frac{B \sigma_s T_c^2}{\log(CE_c/E_s)}$$

where $\sigma_s$ is the bulk flexural strength and $B, C$ are calibration constants from experiment. There are too few data here for a comment regarding the effect of $E_c$ (Fig. 5(a)), but as can be seen from Fig. 6(a), the expected form for the effect of thickness clearly does not fit the data now obtained except perhaps at the highest thicknesses. We are unable formally to account for the discrepancy, although in their experiments they had $30 \leq E_s/E_c \leq 170$, while now it was 9.6, which range difference might be a contributory factor. A more likely source is indenter radius, $r$: Rhee et al. [18] said that for $r \ll T_c$, $F_c$ is independent of $r$, whereas here $r = 20$ mm and $0.6 \leq T_c \leq 2.4$ mm. Clearly a resolution of this point is required, whether this concerns a refinement of the experimental method to account for some overlooked issue, or a refinement of theory. However, we argue that the present conditions better represent the clinical situation and that the results are more likely to be informative in that context.

There are other difficulties in comparing the present results with some others, where discrepancies, which may be caused by differing test conditions, are apparent [12,15, 27,28]. The use of a flat-ended pin on the ceramic surface is one such issue [15]: it is very difficult to align the contacting surfaces exactly parallel, and any premature contact must cause a problem for such brittle materials. In addition, as indicated above, there is necessarily a stress singularity at the edge of the pin even if the alignment is perfect. In other tests, an inappropriately low (2 GPa) [27,28] or high (200 GPa) [12] value for $E_s$ was used. The modulus of elasticity for bulk dentine ($E_d$) has been reported to range from 5 to 20 GPa [29–31] with recent work giving a figure.

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Fig. 7. The tensile stress distribution on a radial section for the three experimental moduli calculated by FEM for 500 N applied load. Disc radius 5 mm, $T_c = 1.0$ mm; $R_c$ is contact radius.
close to 20 GPa for the solid tissue [32]. The crucial point for load-to-failure laboratory tests appears to be the stiffness of the supporting substrate, and as a matter of principle it should have an appropriate modulus of elasticity in order that the clinically-relevant failure mode can be reproduced. It is unfortunate, therefore, that for the present work a suitable substrate material with $E_s \approx 20$ GPa could not be found (which would be a generally useful dentine-mimic material), although, as the discrepancy is only a factor of 2 (as opposed to 10 either way in the cited work), the effect on $S_c$ appears to be relatively small, judging from Fig. 5(c), even though $F_c$ may be more dramatically affected (Fig. 5(a)). However, from the clinical point of view, it is clear that if a tooth is to be built-up before cementing a ceramic crown in place, it seems that it would have to be with a high modulus material so as to avoid a high stress concentration at the cementation surface of the crown and the consequent elevated risk of its failure.

The increase of $F_c$ with $E_s$ while the value of $S_c$ declines (Fig. 5), is in agreement with results reported by Wakabayashi et al. [24], although they used only two substrate materials, as well as with those of Lee et al. [28]. According to the latter, the crack was initiated at the cementation surface of the ceramic for $E_c/E_s > 5$. The minimum value for a dental ceramic is $E_c \approx 70$ GPa, but more typically $> \approx 100$ GPa (Empress II has
Increasing the cement thickness up to 0.025 mm (Table 2); this criterion would therefore generally be satisfied even for the stiffest dentine, consistent with the clinical observation that full-ceramic crowns failed at the cementation surface [1,4,9]. Note that the trend of the value of the failure load for the initial failure of ceramic at the cementation surface varies with the modulus of elasticity of the substrate. On the other hand, since the dentine is usually much softer (\( E_d \approx 20 \) GPa) than the ceramic (100–200 GPa in dentistry), as well as tougher, it (the dentine) can often arrest such cracks. Thus, the relatively soft substrate may increase the bearing capacity of ceramic restorations, i.e. confer ‘damage tolerance’, by redistributing tensile stresses and thus confine the crack within the ceramic. The dentine–enamel junction (DEJ) is believed to play a similar role in arresting cracks, preventing catastrophic failure of teeth [33, 34]. This effect has been observed with microcracks confined to enamel [34].

It follows that the bond strength of the cement should be enough to prevent delamination and thus allow stress transfer from ceramic to tooth; failure to achieve this will lead to the stress in the ceramic being higher than expected. This is one reason for a high risk of ceramic failure with low bond strength cements, such as zinc phosphate [35]. In addition, as Chai et al. [36] pointed out, the cement may also have a role in preventing crack propagation into the substrate, being even weaker and more ductile. In the present work, the normal stress across the ceramic–substrate interface was compressive on the load axis; delamination does not usually occur in such a system. Thus, from previous results [31,33] and the present experiments, it is suggested that increasing the bond strength for ceramic restorations, so that effective stress transfer can occur, may be more advantageous than merely increasing the flexural strength of the ceramic.

A cement film thickness of 0.025 mm is commonly considered ideal, while 0.1 mm is thought to be more realistic and accepted clinically [37]; however, a range of 0.10–0.16 mm has been found [38,39]. It has been reported that increasing the cement thickness up to 0.07 mm decreases the fracture resistance of ceramic crowns [40], but Scherrer et al. [41] reported that this does not occur if the value is kept below 0.3 mm. This discrepancy needs to be resolved, but it remains essential to be aware of the possibility of an effect and so design model systems realistically.

According to clinical practice, the amount of occlusal tooth reduction required for ceramic crowns is usually very limited for premolar and molar teeth (not greater than about 2.0 m). It was found that \( S_c \) depends linearly on \( T_c \) (Fig. 6(c)). This contrasts with the clinical observation that the long-term survival of glass–ceramic restorations does not relate to their thickness [42]. It can only be speculated that other factors dominated the outcome, thereby masking the effect of thickness.

The model system used here has rather obvious differences from the clinical situation in terms of morphology. Nevertheless, the concordance of the behavior in the two circumstances is encouraging in that it may allow a far more relevant context for investigating the effect of many factors related to both ceramic and cement. It is probably only a matter of validating the choice of specimen element radii and substrate thickness to enable the use of the model and mode of testing for standardization and compliance testing.

5. Conclusions

Hertzian indentation offers a means of investigating the behavior of ceramic–cement–substrate structures and as such is highly relevant as a valid simulation of clinical service conditions, if test conditions are appropriately set. These conditions include substrate modulus of elasticity, contact circle radius and cementation. Further study of dental ceramics in this test modality is feasible, for example, of the design of veneer-core bilayer structures and of fatigue behavior.

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References
