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Fracture strength of two oxide ceramic crown systems after cyclic pre-loading and thermocycling

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SUMMARY **The aim of the present study was to investigate the fracture resistance of zirconia crowns and to compare the results with crowns made of a material with known clinical performance (alumina) in away that reflects clinical aspects. Sixty crowns were made, 30 identical crowns of alumina and 30 of zirconia. Each group of 30 was randomly divided into three groups of 10 crowns that were to undergo different treatments: (i) water storage only, (ii) pre-loading (10 000 cycles, 30–300 N, 1 Hz), (iii) thermocycling (5–55°, 5000 cycles) + pre-loading (10 000 cycles, 30–300 N, 1 Hz). Subsequently, all 60 crowns were subjected to load until fracture occurred. There were two types of fracture: total fracture and partial fracture. Fracture strengths (N) were: group 1, alumina 905/zirconia 975 ($P = 0{\cdot}38$); group 2, alumina 904/zirconia 1108 ($P < 0{\cdot}007$) and group 3, alumina 917/zirconia 910 ($P > 0{\cdot}05$). Total fractures were more frequent in the alumina group ($P < 0{\cdot}01$). Within the limitations of this *in vitro* study, it can be concluded that there is no difference in fracture strength between crowns made with zirconia cores compared with those made of alumina if they are subjected to load without any cyclic pre-load or thermocycling. There is, however, a significant difference ($P = 0{\cdot}01$) in the fracture mode, suggesting that the zirconia core is stronger than the alumina core. Crowns made with zirconia cores have significantly higher fracture strengths after pre-loading.**

KEYWORDS: **dental ceramics, dental porcelain, all-ceramic crowns, aluminium oxide, zirconium-dioxide, thermocycling**

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Introduction

A need for non-metallic restorative materials with optimal aesthetics and characteristics such as biocompatibility, colour stability, high wear resistance and low thermal conductivity are often forwarded as reasons for the use of ceramics in dentistry (1, 2). Ceramics are, however, brittle materials because of atomic bonds that do not allow the atomic planes to slide apart when subjected to load. Thus, ceramics cannot withstand deformation of >0·1% without fracturing (3). Furthermore, ceramics in general have pre-existing flaws that vary in both type and size. Such imperfections act as starting points for crack formation whenever ceramic constructions are loaded above a certain level (4). Consequently, when ceramics are used in dental reconstructions, it is important to address these inconveniences when deciding which ceramic systems or designs to employ.

Several new materials and techniques have been introduced in the last 20 years to meet the requirements for use in the oral cavity. Ceramics have now, by and large, been developed to a point where strength and toughness fulfil the demands for use in, e.g. all-ceramic fixed partial dentures (FPDs), although indications for the use of these types of reconstructions are considerably broader today than before (5–7). High-strength ceramics for the cores of crowns and FPDs have been introduced and tested, both *in vitro* and *in vivo*(1, 8–10). Such high strength ceramics based on alumina or

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zirconia, meets the requirements of strength and toughness, which when compared with other ceramics makes them more suitable for use as ceramic cores and copings when extensive loads are expected (1, 11, 12).

Alumina

Aluminium oxide (alumina) has been used for the purpose of increasing strength of dental porcelains for >4 decades (3). Then 15 years ago a new all-ceramic system was introduced, employing a technique where high purity alumina crown copings or FPD cores are fabricated using computer-aided design/computer-aided manufacturing (CAD/CAM) techniques (13, 14). CAD/CAM makes it possible to use industrially manufactured ceramic materials with highly defined quality; to store all production steps electronically; and to attain good reproducibility, accuracy and precision. All these measures are considered important, especially when working with ceramics (15). Subsequent to CAM, the alumina substructures are densely sintered and veneered with dental porcelain to create the appearance of a natural tooth(2). Clinical studies have indicated that such alumina-based crowns may be used for crowns in all locations of the oral cavity (2, 16). Yet, the best mechanical properties of all the dental ceramics are attained with yttrium-stabilized zirconiumdioxide (12, 17).

Zirconia

Zirconiumdioxide (zirconia) is well known as an orthopaedic implant material and has been used in hip surgery for many years (18). By adding a small amount of Y_2O_3 to ZrO_2, it is possible to stabilize the ceramic in a tetragonal phase that normally is unstable at room temperature. Several studies have indicated that flexural strength values of 1200 MPa and fracture toughness values of 9 MPa $m^{1/2}$, which are possible with zirconia, are substantially higher than for other ceramics and that this material therefore could be used for highly loaded, all-ceramic restorations. Hence, suggestions have been made that zirconia could also be a viable alternative to metal in reconstructive dentistry, especially for crowns in the molar region and FPDs (12, 15, 19).

A new, strong, all-ceramic alternative that is based on yttrium-stabilized zirconiumdioxide has been developed for heavily loaded restorations. The material is based on the same technology as the alumina system described above, but the core is made of yttrium-stabilized zirconia instead of alumina. The copings made of zirconia are veneered with a specially developed porcelain.

In the search for new ceramics with improved strength and long-term clinical performance, it is essential to address the ability of the material to resist slow crack growth. Although the properties of zirconia are promising, the tooth-restoration unit forms a laminate system consisting of many layers; (i) *veneer material*, (ii) *the interface between core and veneer* (the compatibility between those two materials), (iii) *core material*, (iv) *the cement and its interfaces* and (v) *the abutment tooth*. When loading such complex multilayered system in the clinical situation it is subjected to fatiguing stresses. As the response to such stresses differs between different laminates, it is not possible to extrapolate strength data from one material system to another. It is therefore important to investigate the fracture resistance of zirconia and to compare the results with a material with known clinical performance (alumina) in a way that reflects clinical aspects as nearly as possible regarding test specimens, environmental influences and test mode. The aim of the present study therefore was to evaluate the strength of one zirconia and of one alumina crown system in a standardized way by mimicking the clinical situation with cyclic mechanical pre-loading and thermocycling under the null-hypothesis that there is not any difference between the fracture resistance between crowns with a core of alumina or of zirconia.

Materials and methods

Sixty crowns designed as stylized norm crowns were to be made for the study, 30 identical crowns of alumina* and 30 of zirconia (Procera®Zirconia*). Each group of 30 was randomly divided into three groups of 10 crowns that were to undergo different treatments according to a test protocol.

Producing crown copings

A master die resembling a molar crown preparation was made of die stone† for the production of 60 crown

*Procera®Alumina; Nobel Biocare, Gothenburg, Sweden.
†Vell-mix; Kerr, Romulus, MI, USA.

copings. The master die was scanned once with a mechanical CAD/CAM scanner (Procera Scanner 40*) and the scanned data were sent via the World Wide Web to the Procera® manufacturer who subsequently produced and delivered the crown copings.

Building up the porcelain veneer

A replica of the master die was cast in a metal alloy‡ to hold and support the crown copings in a reproducible position during porcelain build-up. The shape and dimensions of the veneer build-up were determined by using a specially made knife to shape the porcelain in a standardized way, according to a modification of the technique described by Yoshinari and Dérand (20; Fig. 1).

AllCeram™ porcelain* was used as veneer material for the alumina crowns and Cercon-Ceram S™§ was used for the zirconia crowns, both according to the manufacturer's recommendations (Fig. 2).

In each case, one liner firing and two dentine firings were carried out. Before the dentine was fired a second time, the porcelain was blasted with 50 μm Al_2O_3 under two bars of pressure. In the last step, the crowns were autoglazed. All firing cycles were made according to the manufacturer's recommendations in a calibrated porcelain furnace¶. The furnace was calibrated as recommended by the manufacturer, which briefly means that a wedge-shaped porcelain build-up is fired. If the wedge turns clear during firing, this indicates that it is fired in an accurately calibrated furnace.

Cementation

The norm crowns were to be cemented on separate dies that were specially made by moulding an inlay pattern resin** in 60 A-silicone impressions made of the master die††. All crowns were cemented to the Duralay dies using zinc phosphate cement‡‡ under a standardized load of 15 N for 5 min. Excess cement was removed, and the crowns were stored in distilled water with a temperature of 37 °C until they were subjected to different treatments according to the following test protocol (Table 1).

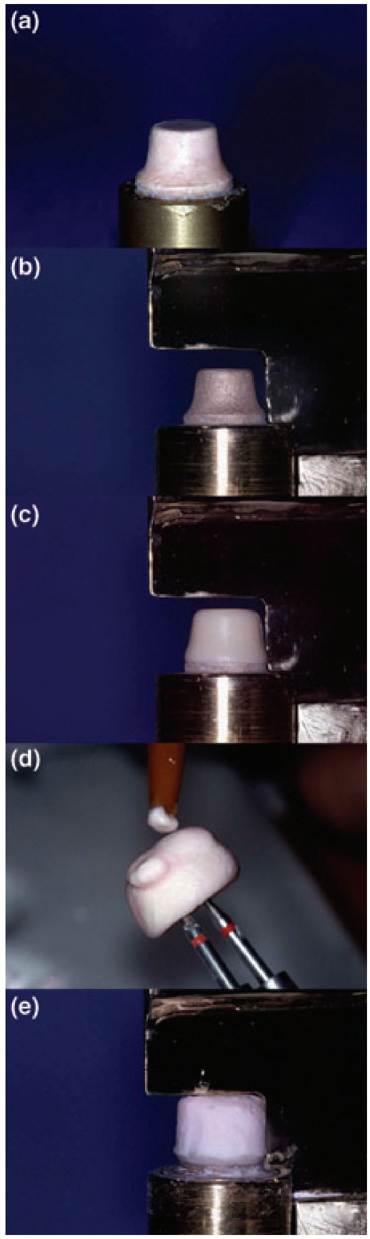

Fig. 1. Building up the porcelain veneer. (a) The master die. (b) Metal replica (crown-holder) and the knife. (c) An alumina-core positioned on the metal-replica. (d) Porcelain build-up. (e) Shaping of the porcelain by aid of the knife prior to firing.

Group 1: control

The crowns in group 1, 10 alumina and 10 zirconia, were stored in distilled water during the test period. The time in water was 7 days – equal to the crowns in

‡Hereaus Kulzer TM; Hereaus Kulzer, Hannau, Germany.
§DeguDent, Hannau, Germany.
¶Dekema Austromat 3001, Dekema Keramik-ösen GmbH, Freilassing, Germany.
**Duralay™; Reliance Dental MFG Co., Worth, IL, USA.
††President, Coltene AG, Altstätten, Switzerland.
‡‡DeTrey zink, Dentsply, Konstanz, Germany.

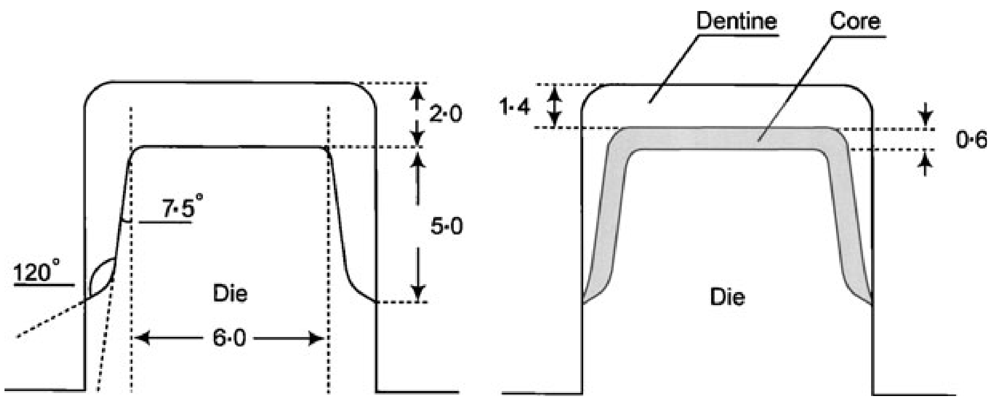

Fig. 2. Design and dimensions of the norm-crowns.

Table 1. Description of test groups and number of crowns in each group

Core material	Group 1 Water storage only	Group 2 Pre-loading (10 000 cycles, 30–300 N, 1 Hz)	Group 3 Thermocycling 5–55°, 5000 cycles, 60 s per cycle + Pre-loading (10 000 cycles, 30–300 N, 1 Hz)	All crown Subsequent to pre-treatment, load until fracture occurred. Cross head speed: 0·255 mm min^{-1}. Steel ball ∅ 2·5 mm
Alumina	10	10	10	30
Zirconia	10	10	10	30

groups 2 and 3, and no crowns were stored under dry conditions during this time.

Group 2: pre-loading

The crowns in group 2, 10 alumina and 10 zirconia, underwent pre-loading in a cyclic pre-loading procedure. Each crown underwent 10 000 cycles at loads between 30 and 300 N with a load profile in the form of a sine wave at 1 Hz. Force was applied with a 2·5-mm diameter stainless-steel ball placed on the occlusal surface of the crowns. All crowns were stored in distilled water during pre-load and mounted in a 10° inclination relative to the long-axis of the crowns (Fig. 3).

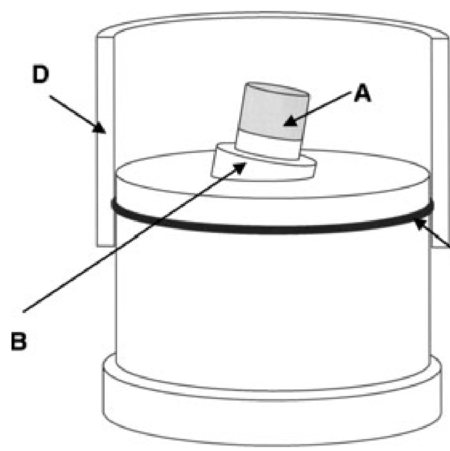

Fig. 3. Jig used for pre-loading and loading tests. A, Norm crown mounted in the jig; B, Brass foundation; C, Rubber gasket and D, Brass cylinder for water storage (shown cross-sectioned).

Group 3: thermocycling

The crowns in group 3, 10 alumina and 10 zirconia, underwent 5000 thermocycles prior to the pre-loading procedure in a specially constructed thermocycling device. Two water baths – 5 and 55 °C – were used. A small basket that could hold 10 crowns on their dies was used to cycle the crowns between the two baths. Each cycle lasted 60 s :20 s in each bath and 10 s to complete the transfer between baths.

Load until fracture

All 60 crowns were individually mounted in a testing jig at a 10° inclination relative the long-axis of the

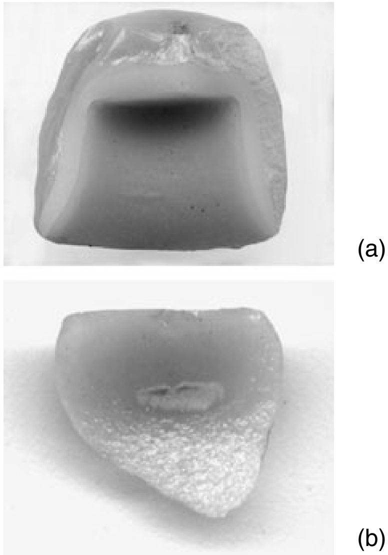

Fig. 4. (a) Illustrates a total fracture of an alumina crown where the fracture surface is clearly visible through both core and veneer materials. (b) Illustrates a chip-off fracture of a zirconia crown. The opaque area in the centre of the fracture surface is a part of the porcelain liner.

crowns, as described above, and finally loaded until fracture occurred using a universal testing machine[§§]. The load was applied with a 2·5-mm diameter stainless-steel ball placed on the occlusal surface of the crowns and a crosshead speed of 0·255 mm min^{-1}. Fracture was defined as occurrence of visible cracks in combination with load drops and acoustic events or by chipping. The loads at fracture were registered, and differences between the groups were calculated using Students' *t*-test. Any differences in fracture mode were calculated using Fisher's exact probability test.

Results

There were two types of fracture: total fracture, through both core and veneer and partial fracture, through the veneer only. Total fractures were more frequent in the alumina group compared with the zirconia group, and this difference was statistically significant ($P < 0{\cdot}01$). In all instances of partial fracture, the fracture was cohesive within the veneer material. Crowns made with zirconia cores showed significantly higher fracture strengths after pre-loading compared with crowns made with alumina cores ($P < 0{\cdot}007$; Fig. 4).

[§§]Instron 4465, Instron, Canton, MA, USA.

During thermocycling seven of 20 crowns showed loss of retention, four alumina and three zirconia. These seven crowns were neither separated from the dies nor recemented before loading to fracture. The other 13 crowns that had undergone thermocycling exhibited no signs of such losses. There was, however, no statistical difference in fracture strength between crowns that exhibited loss and crowns that did not ($P > 0{\cdot}05$; Table 2).

Discussion

It has been suggested that test specimens should have the same critical flaws as crowns made for clinical use and that environmental influences should be reflected in the laboratory settings (12, 21). The approach chosen in the present study was considered justified as the study design took aspects regarding test specimens, environmental influences and test mode into account.

Test specimens

As the manufacturing procedures, recommendations concerning tooth preparation design, dimensions, and shape of both the zirconia and the alumina crowns are identical, it is possible to make comparisons between the two material systems. In both cases, the cores were produced as if they were being made for clinical use. The veneer porcelain is fired according to the manufacturer's recommendations, with appropriate dimensions and an identical, layered build-up technique. Cementations were made according to the manufacturer's recommendations, with zinc phosphate cement, on dies made of Duralay inlay pattern resin, which resembles the modulus of elasticity of dentine (22).

Environmental influences

To assess strength and toughness, some kind of fatigue and aging test must be used. It has been described that ceramic materials undergo an abrupt transition of damage mode and strength degradation after multi-cyclic loads compared with static loading tests (23). Furthermore, the fatigue test should be performed in water as stress corrosion enhances crack growth when water is present at the crack tip (24). When tension and compression periodically occur at the crack tip as a result of load cycles, the damage is increased by access to water. Cyclic pre-load in an aqueous environment

Table 2. Fracture strength and mode [ratio of number of total to partial fractures (total fracture: partial fracture)]

	Group 1		Group 2		Group 3	
Core material	Fracture strength (N)	Fracture mode ratio	Fracture strength (N)	Fracture mode ratio	Fracture strength (N)	Fracture mode ratio
Alumina (mean)	905	8:2	904	9:1	917	9:1
Alumina (s.d.)	104		91		179	
Zirconia (Mean)	975	2:8	1108	6:4	910	3:7
Zirconia (s.d.)	223		190		215	
P-value	0·38	0·01	<0·007	>0·05	>0·05	<0·01

was performed to mimic such aging during service in the mouth and the number of cycles was chosen to make possible comparisons with other studies from our group (20, 22).

Thermocycling is another way to expose materials to fatigue and to simulate aging of the retentive system of crowns and other dental restorations (25). The abrupt change in temperature when specimens are submerged into baths creates stresses in the specimens and especially in the zones between different materials as anisotropy in thermal expansion as well as conductivity results in interfacial stresses. Thermocycling has been found to have a negative influence on cements in general (25, 26). This could explain why seven of 20 crowns exhibited loss of retention during thermocycling. Zinc phosphate, being a brittle cement, was unable to withstand the shear and tensile forces to which it was subjected during the thermocycling fatigue test. These seven crowns were, however, loaded until fracture without recementation considering lack of evidence that those crowns showing no loss of retention in fact might have lost their retention partially. Supposedly, this measure had no negative effect as there was no statistical difference between crowns with or without signs of losses.

The test mode

Ceramic structures tend to fail because of surface tension, where cracks and flaws propagate by slow crack growth to a point where the applied load exceeds the load carrying capacity of the remaining sound portion of the structure, leading to catastrophic failure (27). If a crown is supported by a die made of a high modulus material, the fracture strength will increase dramatically compared with that of crowns supported by a low modulus material. This increases the probability that the load will result in an indention damage at the loading site rather than reflect a fracture mode as seen in clinical failures (21, 28). In the clinical case, there could be a deflection in the dentine, followed by radial expansion in the dentine core and the cervical part of the core as a result of wedging of the crown 'thimble'. Cervical expansion is partly dependent on the cervical preparation mode (chamfer versus shoulder; 22, 29, 30, 32, 33). Secondary to expansion in the dentine, tension can occur in the inner surface of the crown. Duralay, in contrast to stiffer materials, has properties that resemble dentine in this respect (22).

The differences in fracture strength between the two groups that were stored in water only were non-significant. In fracture mode, however, the zirconia cores seemed to be more fracture resistant compared with alumina ones as significantly more of the fractured alumina crowns were totally fractured. This could imply that the zirconia core resists higher loads than alumina ones but that the veneer porcelain fractures at a lower load. A ceramic laminate will always form a constant strain system because of a mismatch of the modulus of elasticity across the core–veneer interface (34). Furthermore, the interface is an important source of structural flaws (35) because of wettability factors – in this study, different surface properties of the two core materials – causing difficulties in building up a dense and homogenous layer of green porcelain, without trapping air bubbles, over the core surface prior to firing.

Veneer fractures often occur during interfacial stresses (28) or because microstructural regions in the porcelain are mechanically defective. Such microstructural flaws include porosities, agglomerates, inclusions and large grained zones (36). As the porcelain was layered under the same environmental conditions and with the same technique in both groups, interfacial stresses rather than mechanically defective microstructural regions in the porcelain are most likely the cause of the higher proportion of veneer fractures in all three zirconia groups compared with the three alumina groups.

The highest strength value was found in the zirconia group that where subjected to pre-load only; the values are significantly higher than those of the other groups. The explanation to this might be the transformation toughening capacity that this material possesses. Suggestions have been made that an increase in fracture toughness of zirconia, induced by external stresses such as impact can be found following phase transformation that occurs in the material when subjected to loads above a certain level (37). Hence, the strength could increase compared with the virgin material. Zirconia's transformation toughening capacity could on the other hand be detrimental to the bond between the core and the veneer. If the core exhibit quasi-plastic yield with deformation over the core–veneer interface, and if the deformation exceed the elastic capacity of the veneering porcelain, then the bond will brake with subsequent loss of the veneer. In the alumina case, however, where the cores do not have this capacity, no yield would take place. But as the strength of alumina is lower compared with zirconia, the crown might fail completely at a lower load. The mechanisms behind those phenomenons are, however, not yet proven and must be further investigated.

The reason that the thermocycled zirconia crowns show no increase in strength, as do the zirconia crowns that where subjected to pre-load only, is probably a result of the deterioration of retention and subsequent to changes in crown support following thermocycling. Such loss of retention has been confirmed in other studies (25) and question that arises is whether the performance of zirconia crowns cemented with resin cements would have improved after thermocycling, as did the performance of the zirconia crowns that where subjected to pre-load only. Further studies are needed to answer these questions.

Conclusions

Within the limitations of this *in-vitro* study, it can be concluded that:

1 There is no difference in fracture strength between the two material systems if crowns are subjected to load *without* any previous cyclic pre-load or thermocycling, but there is a significant difference in the fracture mode, confirming that the zirconia core material is stronger than the alumina core material.

2 Crowns made with zirconia cores have, however, significantly higher fracture strength after pre-loading compared with crowns made with alumina cores. The mechanisms behind this phenomenon are not fully understood, and further studies are needed to confirm the finding.

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