Fracture load and marginal fit of shrinkage-free ZrSiO₄ all-ceramic crowns after chewing simulation

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SUMMARY The aim of this in vitro study was to evaluate the fracture load and marginal accuracy of crowns made from a shrinkage-free ZrSiO₄ ceramic cemented with glass-ionomer or composite cement after chewing simulation. Thirty-two human mandibular molars were randomly divided into two groups. All teeth were prepared for and restored with shrinkage-free ZrSiO₄ ceramic crowns (Everest HPC®, KaVo). The crowns of group A (N = 16) were luted to the teeth using KetacCem® and group B (N = 16) were adhesively cemented using Panavia®21EX. Measurements of the marginal accuracy before and after cementation were made using replicas and an image analysis system. All specimens were exposed to 1.2 million cycles of thermomechanical fatigue in a chewing simulator. Surviving specimens were subsequently loaded until fracture in a static testing device. Fracture

loads (N) were recorded. All specimens survived chewing simulation. The mean fracture loads (±s.d.) were Group A, 1622 N (±433); group B, 1957 N (±806). There was no significant difference between the two groups (P > 0.05). The marginal gap values before cementation were (mean ± s.d.): Group A, 32.7 µm (±6.8); group B, 33.0 µm (±6.7).The mean marginal gap values after cementation were (±s.d.): Group A, 44.6 µm (±6.7); group B, 46.6 µm (±7.7). The marginal openings were significantly higher after cementation for both groups (P < 0.05). All test groups demonstrated fracture load and marginal accuracy values within the range of clinical acceptability. KEYWORDS: ceramic crowns, ZrSiO₄ ceramics,

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Introduction

Many alternatives have been suggested for restoring lost tooth structure in the posterior region. In the 20th century, porcelain-fused-to-metal (PFM) restorations have accounted for a significant proportion of posterior tooth restorations. But perceived aesthetic demands have led to the development of metal-free restorative systems. Furthermore, metal-based crowns have some other disadvantages, such as galvanic and corrosive side effects (1) and gingival discolouration (2). In the last few years, several new all-ceramic systems, which offer comparable stability to PFM, good aesthetics, and simplified fabrication procedures, have been introduced. However, all-ceramic restorations are sensitive to tensile stresses.

More recent fabrication techniques involve the computer-aided manufacturing (CAM) of computer-aided design (CAD) restorations (e.g. Cerec*, DCS[†], Cercon[‡]). Aluminum-oxide- or zirconium-oxide-based ceramics have higher flexural strength and toughness than other commercially available dental ceramics and are popular for CAD/CAM restorations (3). However, the machining of restorations from sintered ZrO₂ is quite time

*Sirona, Bensheim, Germany. [†]DCS Dental, Allschwil, Switzerland. [‡]Degudent, Hanan, Germany. consuming; the machining process for a single unit restoration often takes 2–3 h (4). Evidence shows that the machining process also has a weakening effect on the microstructure of the material. The induced microcracks reduce the reliability of restorations machined from fully sintered ZrO_2 (5, 6). An alternative to slip casting or grinding of sintered blocks is CAD/CAM machining of pre-sintered blocks or blanks at the green stage. The use of a conventional ceramic material process requires an enlarged green body to compensate for the linear sinter shrinkage. The precision of the white body after sintering directly depends on a constant quality of the green bodies and the stability of the sintering process.

These problem areas can be circumvented if a shrinkage-free ceramic material is used. With a so-called reaction bonding process, this is achieved through the volume expansion of one component during the sintering process compensating the volume loss of another. Using a low loss binder, the sinter shrinkage can be further reduced. Such a system based on zirconium silicide (ZrSi₂), zirconia (ZrO₂) and a polymethylsilsesquioxane (PMSS) has been described in detail elsewhere (7). The ceramic based on this system is zircon (ZrSiO₄). It can be moulded to blanks using axial or isostatic pressing. The blanks are stable and can be machined using commercial CAD/CAM equipment.

The Everest[®][§] technique used for the fabrication of the crowns in this study is a combination of the direct– indirect restoration concept (8). There are four modules in the control software: scan, surface, CAD and CAM. After a 3D scan of master model of the abutment tooth, the design of the crown can be produced either on the computer or a wax model of the crown can be scanned (double scan technique, accuracy 20 µm). These two data sets are imported into the CAM module, which calculates the cutting data, taking into account processing properties specific to the material being used. The CNC system (KaVo Everest® Engine[§]) is a computerized five-axis cutting and grinding machine.

The aim of this *in vitro* study was to measure the fracture load and the marginal accuracy of crowns made from a new, shrinkage-free $ZrSiO_4$ ceramic (HPC) cemented with either glass–ionomer or composite cement after exposure to thermo-mechanical fatigue simulating 5 years of clinical service. The null hypothesis for this *in vitro* study was that there is no

statistically significant difference in fracture load and marginal accuracy between crowns which are cemented using two different cements.

Materials and methods

Thirty-two caries-free human mandibular first molars were used for the experiment. The teeth were obtained directly after extraction, cleaned and stored in 0.1% thymol solution at room temperature throughout the study. All teeth were examined under a light microscope to detect cracks before including them in the study. An impression of the teeth was made with Formasil[®] Xact[¶] which was helpful for the fabrication of the ceramic restorations so as to restore the original shape of the tooth. All teeth were prepared for allceramic full-coverage crowns with a circular shoulder. An occlusal reduction of 1.8-2.0 mm was prepared, followed by a circular 1.2-mm wide shoulder with a diamond bur (80-µm grit). The circular finishing line was 1 mm above cemento-enamel junction. The final preparation was made with a diamond bur (30–40-µm grit), and all sharp angles were rounded. So as to imitate physiological tooth mobility, all roots of the selected teeth were covered with an artificial periodontal membrane made out of gum resin of a 0.25-mm thickness (Anti-Rutsch-Lack®**). The teeth were embedded in sample holders at an inclination of 90°, using auto polymerizing resin extended to a level 2 mm below the cemento-enamel junction (Technovit 4000®⁺⁺).

Impressions of the abutment teeth was made with a vinyl polysiloxane impression material (Monopren®^{‡‡}) and then master models were poured using Everest Rock® dental stone[§]. Afterwards, using the silicone key from the models before preparation as a guide, full wax-ups of the crowns were performed (Everest® Wax Set[§]). All models and the wax-ups were sent to the manufacturer (KaVo), so as to fabricate the all-ceramic crowns using the double scan technique. Thirty-two crowns were manufactured using the Everest HPC ZrSiO₄ ceramic. The crowns were milled in Everest Engine CAM milling unit in the green stage. Subsequently, a tempering process at a temperature of

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Table 1.	Overview of	the cement	s in the two	groups of	specimens
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Group	Cement	Brand name
A	Glass–ionomer cement	KetacCem®
B	Autopolymerizing composite cement	Panavia 21EX®

1575 °C (4 h) was performed to sinter the green bodies.

The measurements of the marginal accuracy before and after cementation were made using an epoxy replica. For this purpose, the crowns were seated on their respective abutment teeth and fixed by finger pressure. An impression of the marginal areas around the crowns was taken with a vinyl polysiloxane impression material (Dimension Grant $L^{\otimes S}$), and each impression was poured using epoxy resin (Epoxydie $\mathbb{S}^{\mathbb{T}}$). For measurements after cementation, the replicas were made in the same manner.

The teeth were randomly divided into two groups of 16 samples each (Table 1). The ceramic crowns of group A were cemented on the abutment teeth using glassionomer cement (KetacCem®^{SS}) and group B using autopolymerizing composite cement (Panavia®***). The inner surfaces of all of the crowns were sandblasted before cementation was performed.

A computer system consisting of a stereomicroscope⁺⁺⁺, a 3CCD camera^{‡‡‡} and a personal computer (IBM-Compatible Personal Computer with Microsoft NT Operating System 4.0) with a special software (Analysis® 3.0^{§§§}) were used to record the measurements. The 3CCD camera produced a ×40 magnification on a high-resolution $(800 \times 600 \text{ pixel})$ computer monitor, so that a video image of the marginal gap could be examined. The measurements of the marginal discrepancies around the circumference of the tooth were made on-screen. The gap between the outermost edge of the suprastructure and the preparation line was defined as the standard for marginal accuracy. Sixtyfour single measurements were made around the circumference of each specimen (9). The mean value of marginal discrepancy was calculated for each specimen, and this value was used to determine the median



Figure 1. Fracture load test of an all-ceramic crown. The load was applied to the occlusal surface of the specimens.

marginal discrepancy for each group before and after cementation of the ceramic crowns.

All specimens were loaded in a computer-controlled dual-axis chewing simulator^{¶¶¶} to 1·2 million cycles of thermo-mechanical fatigue to simulate 5 years of clinical service. The applied load was 49 N. A 6-mm-diameter ceramic antagonist Steatit® ball**** was applied vertically onto the occlusal surface of the restorations with a frequency of 1·3 Hz (10). The thermo-cycling was 5–55 °C for 60 s each with an intermediate pause of 12 s, maintained by the thermo-statically controlled liquid circulator^{††††}. Specimens that survived the dynamic loading were examined under a light microscope for fractures of the teeth or of the crowns.

Then all specimens were loaded until fracture occurred using a universal-testing machine^{$\pm\pm\pm\pm$}. Two layers of a 0·3-mm-thick tin foils were placed over the occlusal surface of the teeth to achieve a homogenous stress distribution. A perpendicular load was applied to the occlusal surface of samples (Fig. 1) under a stroke control of 2 mm min⁻¹. The loads required for fracturing the specimens were recorded.

The statistical analysis of the fracture load tests was performed using pairwise comparisons with the Wilcoxon rank test and using paired *t*-tests for the marginal accuracy analysis. A significance level of P < 0.05 was used for all comparisons.

- ****Höchst Ceram Tec, Wunsiedel, Germany.
- ⁺⁺⁺⁺Haake, Karlsruhe, Germany.
- ^{‡‡‡‡}Zwick, Z010/TN2S, Ulm, Germany.

^{§§}3M ESPE, Seefeld, Germany.

^{¶¶}Ivoclar, Schaan, Switzerland.

^{***21} EX, Kuraray, Tokyo, Japan.

⁺⁺⁺Zeiss, Oberkochen, Germany.

^{‡‡‡}Sony, Köln, Germany.

^{§§§}Soft-Imaging Software GmbH, Münster, Germany.

^{¶¶¶}Willytec, Munich, Germany.



Figure 2. Box plot of the static fracture loads (in N) after artificial aging of specimens. The all-ceramic crowns in group A were cemented with a glass isonomer cement (Ketac Cem) and in group b with an autopolymerizing cement (Panavia 21 EX).

Results

All specimens in groups A and B survived the chewing simulation. The fracture loads (mean \pm s.d.) were Group A, 1622 N (\pm 433); group B, 1957 N (\pm 806). The results of the fracture load test are presented as a box plot (Fig. 2). Wilcoxon rank tests showed no significant differences between the fracture load values of the two groups of specimens (P > 0.05).

The mean marginal gap values before cementation were: Group A, $32.7 \ \mu\text{m}$ (± 6.8); group B, $33.0 \ \mu\text{m}$ (± 6.7). The mean marginal gap values after cementation were: Group A, $44.6 \ \mu\text{m}$ (± 6.7); group B, $46.6 \ \mu\text{m}$ (± 7.7). The maximum values after cementation were $59.8 \ \mu\text{m}$ for group A and $62.1 \ \mu\text{m}$ for group B. Further results of the marginal accuracy test are presented in Table 2. The statistical analysis (paired *t*-test) of the marginal accuracy test showed significant differences of marginal gap values between before and after cementation of group A and B (P < 0.05) but not between group differences.

Discussion

In the present study all of the samples have been exposed to the artificial ageing and all the samples survived the test. A computer-controlled dual-axis chewing simulator was used for the artificial ageing. The artificial chewing cycle in the artificial oral environment is designed to correspond as closely as possible

Table 2. Descriptives of marginal accuracy test results in μ m before and after cementation

	Group A: Ketac- Cem		Group B: Panavia	
	Before	After	Before	After
Mean	32·7a	44·6b	33·0a	46·6b
s.d.	6.8	6.6	6.7	7.7
Median	32.8	43.0	33.7	47.8
Minimum	21.2	34.3	23.3	36.3
Maximum	47.0	59.8	47.4	62.1

Mean values with the same letter are not significantly different. A significant difference was found in each before/after cementation comparison for both types of cements (P < 0.05).

to physiological conditions (11, 12). The magnitude, duration and frequency of the force applied are comparable with values reported in the literature (13). Also a thermocycling with temperature changing from 5 to 55 °C was performed. This is often reported by other authors so as to evaluate the long-term success of restorations (12, 14). While this testing procedure is only an approximation, the results suggest that the tested crowns withstand simulated physiological conditions and are thus fit for clinical testing.

Published data on bite forces indicate that the maximum biting force that may occur in the posterior dental area vary between 300 and 880 N (15-17). Few authors have shown higher maximum bite forces of above 4000 N (16). However, dynamic chewing forces are usually lower. In this study, all specimens had fracture load values of >950 N. These values exceeded the maximum limit of natural teeth (500 N) as suggested by Kappert (18). The fracture load found for the ZrSiO₄ crowns in this study was also comparable with fracture load found for crowns made from the pressed ceramic 'Empress 2'. Others have reported similar fracture loads for posterior all-ceramic crowns in the range of 1086 N (Procera) and 1183 N (In-Ceram Zirconia). Unfortunately, there are limitations to the comparability of the results. First, rather large standard deviations were found within the Panavia testing group. This may be related to the variation in tooth anatomy and preparation design of the natural abutment teeth which in turn may have resulted in variations in core thickness. Also, variations in the material properties are a possible source of variation because tests were performed using experimental batches.

Secondly, in our case, full crowns made from the core material only were fabricated, whereas all other crowns are usually made from a core material and a veneer layer. Thirdly, in contrast to previous tests, a set-up using artificial abutments has been used comprising the comparability of the results to other studies (19). Still, it can be concluded that the novel $ZrSiO_4$ ceramic material is fit for clinical use.

As a preparation for cementation, the inner surfaces of the crowns were sandblasted. In group A, the crowns were cemented with a glass-ionomer cement (KetacCem®^{§§}) and in group B they were adhesively cemented to the abutment teeth with chemically polymerizing resin cement (Panavia®21EX***). Although the resin bond to silica-based ceramics is well researched and documented, few in vitro studies on the resin bond to high-strength ceramic materials were identified. In vitro studies have shown that etching with hydrofluoric acid has no influence on the surface structure of ZrO₂ or aluminum oxide and neither on the bond strength of cements to highstrength ceramics (20). The bond strength to zirconium oxide ceramics can be increased by sandblasting and by using a special adhesive monomer (21, 22). To date, there are no data on the bond strength of either glass-ionomer or composite cements to ZrSiO₄ ceramic. As for full coverage crowns, there appears to be no significant influence of the cement on the load to fracture after thermomechanical loading. However, a slightly higher fracture load has been observed for the cementation with Panavia.

Research into the biomechanical behaviour of teeth is generally necessary to maintain the physiological state of specimens as close as possible to that *in vivo*. Extracted human teeth were used in this study, as in several *in vitro* studies, which tested the fracture load of all-ceramic crowns placed on extracted natural teeth (23). For mimicking the physiological mobility of the teeth during chewing simulation and fracture load testing (12), a thin layer of gum resin was painted on the roots of the teeth in the present study. It has been demonstrated that sample mobility is a decisive factor in the evaluation of fracture load (24), and when a small amount of tooth rotation is allowed, failure of the restoration is more relevant to the clinical situation (25).

In the literature, the values of marginal gap vary between 4 μ m for cast metal crowns (26) and over 100 μ m for CAD/CAM crowns made from high-

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performance ceramic (27) A marginal gap of <50 µm has been recommended as clinically acceptable (28, 29). A clinical study of 300 all-ceramic full coverage restorations followed up to 5 years reported a mean marginal adaptation of 30 µm before and after luting of the Al₂O₃-copings onto the tooth. However, the marginal opening was up to 135 µm at the deepest part of the chamfer (30). The mean marginal gap recorded in the present study is in accordance with, or smaller than that reported previously (27). After cementation, the marginal gap of the crowns of both groups was significantly higher than before cementation. This has also been shown in other in vitro studies (23, 31). While the mean marginal accuracy achieved in this study is well within the range proposed by others, the low values may have been achieved at the price of a larger internal misfit. It has been shown previously that small marginal openings correlate with larger internal gap values of 100-200 µm in vitro (32) as well as in vivo (33). However, even the maximum gap sizes after cementation found for either group do only slightly exceed 60 µm and, therefore, are well below what has been 100 µm. This value has been regarded as the level of clinical acceptability by several authors (32 - 35).

The results of this *in vitro* study appear to be well within the range of clinical acceptability. However, clinical trials are necessary to validate the results.

Conclusion

It can be concluded that

1 The fracture load values of Everest® HPC all-ceramic crowns have exceeded average physiological chewing forces as suggested by the literature and

2 the marginal accuracy of Everest® HPC all-ceramic crowns is comparable with values found for other all-ceramic systems,

3 there is no statistically significant influence of the type of cement on the mean failure load and median marginal discrepancy.

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