Fracture Strength of Zirconia Implants after Artificial Aging

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ABSTRACT

Background: Zirconia (ZrO_2) might be an alternative material to titanium (Ti) for dental implant fabrication. However, no data are available on the fracture strength of one-piece ZrO_2 oral implants.

Purpose: The objective of this study was to evaluate the fracture strength of ZrO_2 implants after exposure to the artificial mouth.

Materials and Methods: One hundred twenty ZrO_2 and Ti implants were used. The Ti implants were divided into two control groups (A and B). ZrO_2 implants manufactured from yttria-stabilized tetragonal ZrO_2 polycrystal (Y-TZP) in group C, from Y-TZP dotted with alumina (Y-TZP-A) in group D, and from Y-TZP-A with a modified surface in groups E and F were used. In group F, the implant heads were prepared, and in group G, the implants were restored with ZrO_2 crowns. Each group included 16 samples with the exception of group D, which included 24 samples.

A subgroup of each implant type (eight implants) was subjected to thermomechanical cycling in a chewing simulator prior to fracture testing. Test specimens were then loaded until a fracture occurred.

Results: Seven of the 120 samples failed in the chewing simulator. ZrO_2 implant fracture occurred at 725 to 850 N when the implants were not prepared, and at 539 to 607 N when prepared. The samples in group A fractured at the level of the abutment screw. All ZrO_2 implants fractured at the level of the Technovit® resin (Heraeus Kulzer GmbH & Co., Wehrheim, Germany). No fracture of the ZrO_2 crowns in group G was observed.

Conclusion: Mean fracture strength values obtained were all within the limits of clinical acceptance. However, implant preparation had a statistically significant negative influence on the implant fracture strength. Long-term clinical data are necessary before one-piece ZrO_2 implants can be recommended for daily practice.

KEY WORDS: artificial mouth, fracture strength, titanium implants, zirconia implants

INTRODUCTION

Replacing single missing teeth, in particular in the anterior region, always presents a challenge. Patients commonly oppose the preparation of intact teeth as abutments for a fixed partial denture. Other treatment options, including resin-bonded restorations, orthodontics, and removable partial dentures, have been proven to be less than ideal, and there is a considerable interest in replacing missing teeth with implant-supported crowns.[1–3] However, using conventional titanium (Ti) implants as abutments for tooth replacements, the grayish color of the Ti implant may often be perceived through the peri-implant mucosa impairing aesthetic outcomes in particular in the presence of a thin mucosal biotype.[4,5] Furthermore, there are reports that metals, including Ti, are able to induce a nonspecific immunomodulation and autoimmunity.[6] Highly sensitive immunologic in vitro tests have demonstrated sensitization to Ti.[7] Clinical implications and relevance of these observations are at present not understood.

Because of potential immunological and possible aesthetic compromises using Ti implants, novel implant technologies, including ceramic implants, are being

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developed, maintaining the characteristics that gave the Ti implants their high success rates.[8] However, ceramics are known to be sensitive to shear and tensile loading, and surface flaws may lead to early failure. This disadvantage made it difficult to apply all ceramics for the fabrication of dental implants. One such implant material, aluminum oxide, was used, for example, with the Tübingen Implant (Frialit I),[9] but because of its insufficient physical properties, it was withdrawn from the market. Therefore, ceramic implants imply a high risk for fracture, and a study on their strength is needed.

Recently, a ceramic biomaterial, zirconium dioxide (zirconia, ZrO_2), with a potential for future use as oral implant material, was introduced. ZrO_2 possesses good physicochemical characteristics including a high flexural strength (900–1, 200 MPa), Vickers hardness (1, 200), and Weibull modulus (10–12).[10] A mechanism known as *transformation toughening* is considered to be the basis for the high strength of yttria-tetragonal ZrO_2 polycrystal (Y-TZP).[11–14] The use of ZrO_2 for the manufacture of oral implants has been suggested.[15] However, there is only limited information available concerning oral ZrO_2 implants. In vitro studies prior to clinical tests are necessary to avoid catastrophic in vivo outcomes such as those observed with the use of alumina implants.[16] The objective of the present preclinical study was to evaluate the fracture strength of one-piece ZrO_2 oral implants after exposure to the artificial mouth where a clinical service of 5 years was simulated.

MATERIALS AND METHODS

One hundred twenty screw-type ZrO_2 and Ti implants were used. The experimental groups shown in Figure 1A included the following:

1. Group A: 16 Brånemark Ti external hex implants (3.75 × 15 mm, MK III Groovy, Nobel Biocare AB, Göteborg, Sweden) restored with a custom-made abutment (Nobel Biocare AB). Subgroup A1: no artificial load, fracture testing; A2: with artificial load, fracture testing. The abutment was attached to the implant using a torque of 35 Ncm.
2. Group B: 16 NobelDirect® Ti implants (3.75 × 15 mm, Nobel Biocare AB). Subgroup B1: no artificial load, fracture testing; B2: with artificial load, fracture testing.
3. Group C: 16 yttria-reinforced, hot isostatically pressed tetragonal ZrO_2 polycrystal Sigma® implants (4.28 × 14.4 mm, Incermed, Lausanne, Switzerland). Subgroup C1: no artificial load, fracture testing; C2: with artificial load, fracture testing.
4. Group D: 24 yttria-reinforced, hot isostatically pressed tetragonal ZrO_2 polycrystal – dotted with alumina – implants (Y-TZP-A BIO-HIP®, 4.3 × 16 mm, Nobel Biocare AB) (see Figure 1B). Subgroup D1: no artificial load, fracture testing; D2: with artificial load, fracture testing; D3: with artificial load, without thermocycling, fracture testing.
5. Group E: 16 Y-TZP-A BIO-HIP (4.3 × 16 mm) implants with a ZiUnite® surface (Nobel Biocare AB). Subgroup E1: no artificial load, fracture testing; E2: with artificial load, fracture testing.
6. Group F: 16 Y-TZP-A BIO-HIP (4.3 × 16 mm) implants with a ZiUnite surface (see Figure 1B). Subgroup F1: 0.5-mm chamfer preparation, no artificial load, fracture testing; F2: 0.5-mm chamfer preparation, with artificial load, fracture testing.
7. Group G: 16 Y-TZP-A BIO-HIP (4.3 × 16 mm) implants with a ZiUnite surface. Subgroup G1: 0.5-mm chamfer preparation, all-ceramic crown, with artificial load, fracture testing; G2: 1-mm chamfer preparation, all-ceramic crown, with artificial load, fracture testing.

The endosseous part of the implants in groups E, F, and G were coated with a slurry containing ZrO_2 powder and a pore former. During the sintering of the slurry, the pore former burnt out and left a porous surface.[17]

The heads of the ceramic implants in groups F and G were prepared with diamonds (large grain: 80 μm, and fine grain: 40 μm) and water cooling. The preparation form of the implant heads was that of a maxillary central incisor (see Figure 1B). In group F, the cervical finish line was a 0.5-mm chamfer. In subgroup G2, the preparation of the cervical finish was a 1-mm chamfer. After the implant preparation in group G, impressions were taken with a polyether impression material (Impregum™, 3M Espe, Seefeld, Germany) and a mini tray. A model of the implant head was created using epoxy resin, which was subsequently scanned using the Procera® technique (Nobel Biocare AB).[18] The implants were restored with all-ceramic crowns. The crowns were fabricated from Procera Zirconia® and veneered with Nobel Rondo® (Nobel Biocare AB). Before the cementation, the intaglio surface of the crowns was airborne-particle abraded (110-μm Al_2O_3 powder at a pressure of

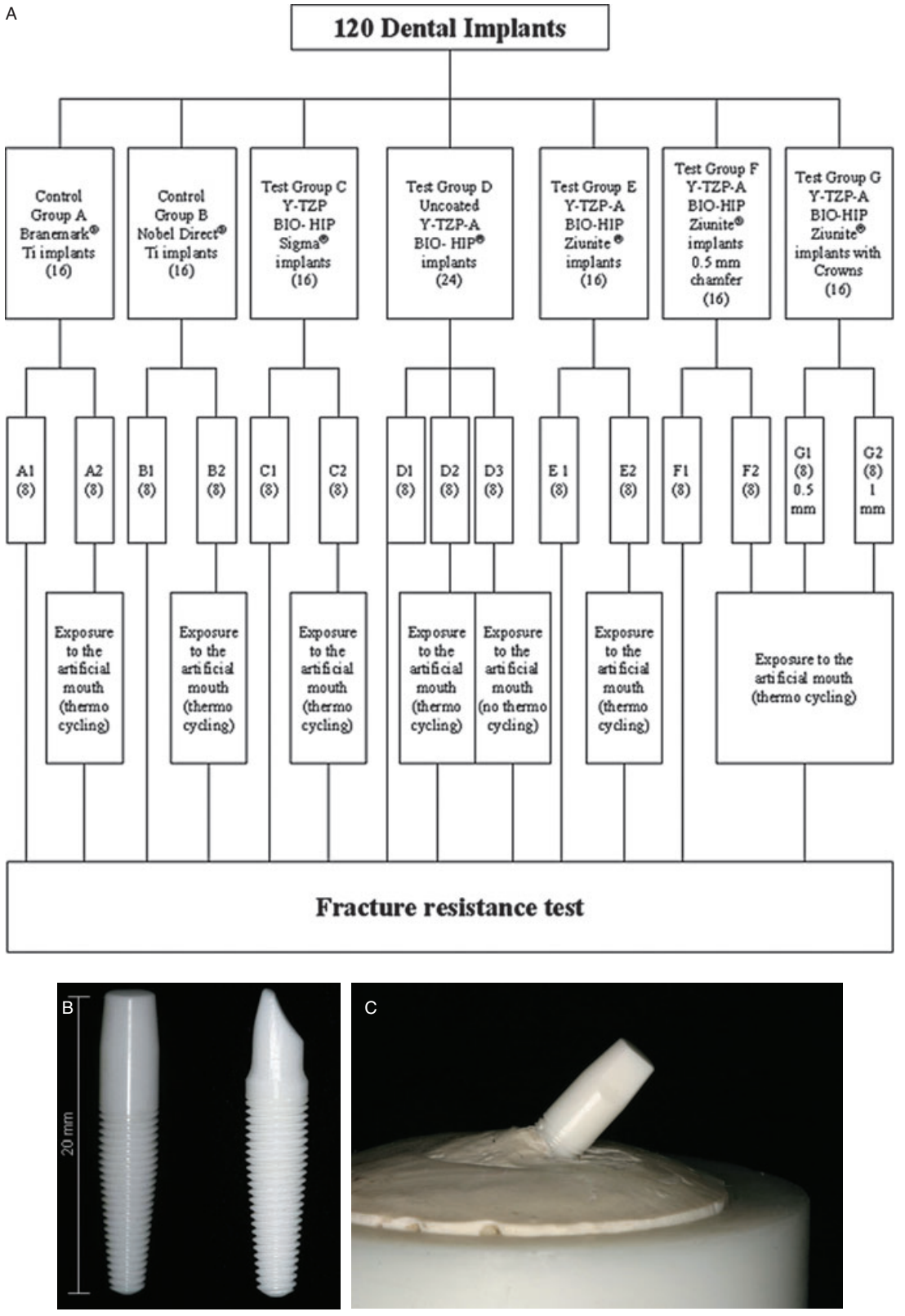

Figure 1 *A,* Outline of the study; *B,* (*left*) an implant of group D (Y-TZP-A BIO-HIP® implant), (*right*) chamfer preparation of a ZiUnite® implant (group F); *C,* a ZiUnite implant embedded in the sample holder (inclination angle of 130°).

2 bars) then treated with a silane coupling agent (Clearfil SE Bond Primer and Clearfil Porcelain Bond Activator, Kuraray, Tokyo, Japan), and finally cemented with Panavia® 21 (Kuraray). The crowns were seated onto the implants with finger pressure. Excess cement was removed, and an airblock (Oxyguard®, Kuraray) was applied until a complete setting of the cement.

Seventy-two specimens (subgroups A2, B2, C2, D2, D3, E2, F2, G1, and G2) were exposed to 1.2 million cycles of thermomechanical fatigue loading in a chewing simulator (Willytech, Munich, Germany). This protocol simulated 5 years of clinical use.[19,20] The implants were therefore placed into special sample holders to the height of the first implant thread and were stabilized with an autopolymerizing acrylic resin (Technovit® 4000, Heraeus Kulzer GmbH & Co., Wehrheim, Germany) at a faciolingual angle of approximately 130° to the horizontal plane, replicating the position of upper central incisors (see Figure 1C).

A force of 98 N (10 kg) was chosen to simulate a load within the clinical range[21,22] and was applied at a frequency of 1.6 Hz using a steatite ball with a 6-mm diameter as antagonist (Hoechst Ceramtec, Wunsiedel, Germany). All specimens with the exception of subgroup D3 were subjected to a simultaneous thermocycling between 5 and 55°C for 60 seconds each with an intermediate pause of 12 seconds controlled by a liquid circulator (Haake, Karlsruhe, Germany).

All specimens that did not fracture during the dynamic loading were loaded until fracture in a universal testing machine (Z010/TN2S, Zwick, Ulm, Germany). A perpendicular load was applied with a flat-surfaced metal rod (ø1 cm) on the angulated implants under a crosshead speed of 2 mm/min at an angle of 130° to the horizontal plane. The fracture loads were recorded on an X-Y writer with the TestXpert® V 7.1 software, with the failure recorded at the first sharp drop-down of the graphical curve (fracture of the ceramic, bending of the Ti).

The statistical analysis was performed with the support of the Institute of Medical Biometry and Medical Informatics, Albert-Ludwigs University, Freiburg, Germany, and included multiple pairwise comparisons using the Wilcoxon rank sum test (The R Project for Statistical Computing, R Foundation for Statistical Computing, Vienna, Austria) at a significance level of 0.05.

RESULTS

Seven of the 120 samples failed in the chewing simulator. Table 1A shows the incidences that occurred in the different subgroups and survival rates.

The fracture strength values are summarized in Table 1B and Figure 2. The specimens that failed in the artificial mouth were considered to have fractured at 98 N. All samples in group A (two-piece Brånemark implants) fractured at the level of the abutment screw. In

TABLE 1A Implant Fracture during Dynamic Loading (Chewing Simulator) and Survival Rate Analysis

Group	Total Number of Implants Used	Failures	Fracture at Cycle	Survival Rate after 1,200,000 Cycles (%)
A2 (control)	8	1 (screw fracture)	475,000	87.5
B2 (control)	8	0	0	100
C2 (test)	8	4	1 implant at 2,000 3 implants at 250,000	50
D2 (test)	8	0	0	100
D3 (test)	8	1	190,000	87.5
E2 (test)	8	1	275,000	87.5
F1 (test)	8	0	0	100
F2 (test)	8	0	0	100
G1 (test)	8	0	0	100
G2 (test)	8	0	0	100

Control A2: Brånemark® Ti implants (exposure to the artificial mouth); control B2: Nobel Direct® Ti implants (exposure to the artificial mouth); test C2: Y-TZP BIO-HIP Sigma® implants (exposure to the artificial mouth); test D2: uncoated Y-TZP-A BIO-HIP® implants (exposure to the artificial mouth); test D3: uncoated Y-TZP-A BIO-HIP implants (exposure, no thermocycling); test E2: Y-TZP-A BIO-HIP ZiUnite® implants (exposure + no preparation of the implant heads); test F1: Y-TZP-A BIO-HIP ZiUnite implants (no exposure + 0.5-mm chamfer); test F2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + 0.5-mm chamfer); test G1: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 0.5-mm chamfer); test G2: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 1-mm chamfer).

TABLE 1B Statistical Analysis of the Fracture Strength Results in Newtons

Group	Minimum	1st Quartile	Median	Mean	3rd Quartile	Maximum
A1 (control)	663	757.95	810	825	897.54	1,001
A2 (control)	98	726.08	785	715	822.77	931
B1 (control)	2,896	4,253.49	7,004	5,717	7,004.93	7,007
B2 (control)	2,162	2,407.58	2,731	2,749	3,104.46	3,441
C1 (test)	1,089	1,202.62	1,328	1,337	1,456.75	1,632
C2 (test)	98	98	724	855	1,528.39	1,941
D1 (test)	804	832.10	954	940	1,024.30	1,102
D2 (test)	761	832.23	863	879	903.49	1,045
D3 (test)	98	904.75	1,018	980	1,210.27	1,479
E1 (test)	711	748.05	769	850	966.27	1,101
E2 (test)	98	737.22	776	725	836.46	978
F1 (test)	530	539.90	563	578	616.47	663
F2 (test)	479	548.14	592	607	634.78	805
G1 (test)	403	508.61	552	542	591.01	638
G2 (test)	464	486.61	511	539	589.98	657

Control A1: Brånemark® Ti implants (no exposure to the artificial mouth); control A2: Brånemark Ti implants (exposure to the artificial mouth); control B1: Nobel Direct® Ti implants (no exposure to the artificial mouth); control B2: Nobel Direct Ti implants (exposure to the artificial mouth); test C1: Y-TZP BIO-HIP Sigma® implants (no exposure to the artificial mouth); test C2: Y-TZP BIO-HIP Sigma implants (exposure to the artificial mouth); test D1: uncoated Y-TZP-A BIO-HIP® implants (no exposure to the artificial mouth); test D2: uncoated Y-TZP-A BIO-HIP implants (exposure to the artificial mouth); test D3: uncoated Y-TZP-A BIO-HIP implants (exposure, no thermocycling); test E1: Y-TZP-A BIO-HIP ZiUnite® implants (no exposure + no preparation of the implant heads); test E2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + no preparation of the implant heads); test F1: Y-TZP-A BIO-HIP ZiUnite implants (no exposure + 0.5-mm chamfer); test F2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + 0.5-mm chamfer); test G1: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 0.5-mm chamfer); test G2: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 1-mm chamfer).

group B (Nobel Direct), only a bending of the one-piece Ti implants was observed. In groups D (Y-TZP-A BIO-HIP implants), E (ZiUnite), F (ZiUnite with 0.5-mm chamfer), and G (ZiUnite with crowns including a 0.5/1.0-mm chamfer), all implants fractured at the level of the Technovit resin. No fracture of the ZrO_2 crowns in group G was observed.

The multiple pairwise comparisons of the fracture strength among the Y-TZP-A implants are shown in Table 2: (1) no significant effect of the exposure of the implants to the artificial mouth: D1 versus D2, E1 versus E2, and F1 versus F2; all: $p > .05$; (2) no significant effect of implant surface treatment: D1 versus E1, $p > .05$; (3) a significant effect of implant head preparation: E1 versus F1, $p > .05$; (4) no significant effect of depth of implant head preparation: G1 versus G2, $p > .05$; and (5) no significant effect of crown coverage: F1 versus G1, and F2 versus G2, all: $p > .05$.

DISCUSSION

The objective of this study was to evaluate the fracture strength of one-piece ZrO_2 oral implants simulating occlusal forces exerted in the oral cavity. In this context, it is necessary to consider that the range of biting forces may vary markedly from one area of the mouth to another, and from one individual to another. In a recent clinical investigation evaluating biting and chewing forces, the maximum bite forces in the posterior dentition ranged from 250 to 400 N, and in the anterior dentition from 140 to 170 N. Normal chewing forces in the posterior dentition ranged from 110 to 125 N, and in the anterior dentition from 60 to 75 N.[22] A cycle load of 98 N was used in this study to simulate physiologic biting and chewing forces in the anterior dentition.

Mean fracture strength values for the various implants that were either exposed or not to the artificial mouth exceeded 400 N. The fracture values are within the limits of clinical acceptance, when compared with the physiologic forces in the oral cavity.[22] The values also exceeded 300 N, which seems to be the minimum fracture strength value proposed for restorations of the posterior dentition.[23,24]

An explanation for the lower survival rate of subgroup C2 (50%) compared with the other subgroups

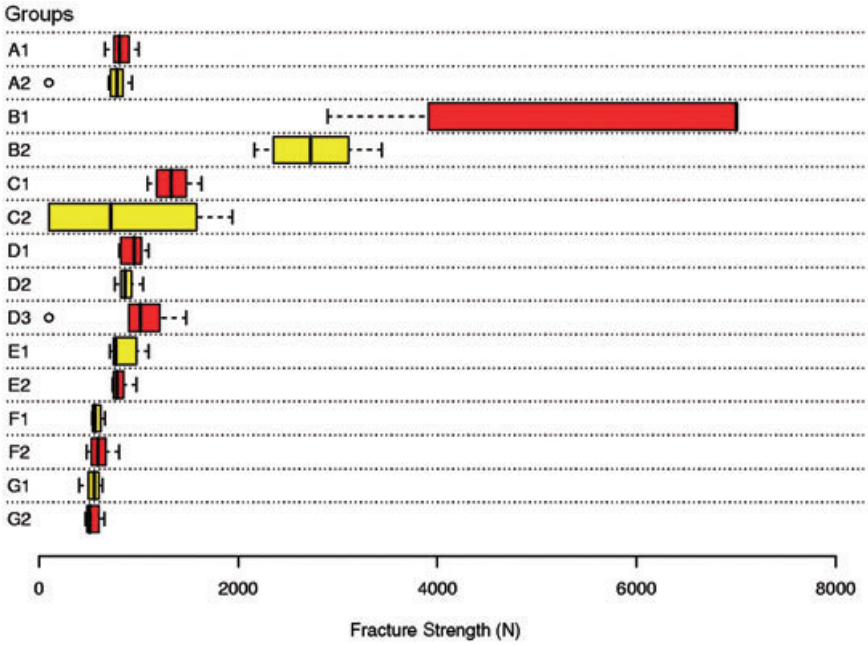

Figure 2 Box plot diagram of the fracture strength results in newtons. Control A1: Brånemark® Ti implants (no exposure to the artificial mouth); control A2: Brånemark Ti implants (exposure to the artificial mouth); control B1: Nobel Direct® Ti implants (no exposure to the artificial mouth); control B2: Nobel Direct Ti implants (exposure to the artificial mouth); test C1: Y-TZP BIO-HIP Sigma® implants (no exposure to the artificial mouth); test C2: Y-TZP BIO-HIP Sigma implants (exposure to the artificial mouth); test D1: uncoated Y-TZP-A BIO-HIP® implants (no exposure to the artificial mouth); test D2: uncoated Y-TZP-A BIO-HIP implants (exposure to the artificial mouth); test D3: uncoated Y-TZP-A BIO-HIP implants (exposure, no thermocycling); test E1: Y-TZP-A BIO-HIP ZiUnite® implants (no exposure + no preparation of the implant heads); test E2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + no preparation of the implant heads); test F1: Y-TZP-A BIO-HIP ZiUnite implants (no exposure + 0.5-mm chamfer); test F2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + 0.5-mm chamfer); test G1: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 0.5-mm chamfer); test G2: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 1-mm chamfer).

could be the quality and microstructural design of the ZrO_2 used for the fabrication of the Sigma implants, as ZrO_2 materials are not the same.[25–27] However, when the mean fracture strength values of the Sigma implants in subgroup C1 (1, 337 N) and the ZiUnite implants in subgroup E1 (850 N) were compared, a statistically significant difference was observed ($p < .05$). These results demonstrate a higher stability and strength of the Sigma implants when they are not exposed to the artificial mouth. The results suggest that Y-TZP without alumina has a higher strength but a lower resistance to degradation.

Implant exposure to the artificial mouth had no statistically significant influence on the fracture strength of the implants manufactured of Y-TZP-A (E1 vs E2, and F1 vs F2; all: $p > .05$). It seems that this material would not significantly age over a period of 1.2 million cycles. To evaluate if a longer artificial load would significantly decrease the fracture strength, another eight implants (F2) were submitted to 5 million cycles (~20 years of clinical function). The resulting fracture strength was not significantly lower compared with the other prepared ZiUnite groups (data not shown).

Coating the Y-TZP-A implants to achieve a porous surface did not significantly alter the fracture strength values (D1 vs E1, and D2 vs E2; all: $p > .05$). The coating procedure is recognized as a safe procedure regarding implant stability, and it has a significantly positive impact on osseointegration and removal torque.[17]

All of the implants that were submitted to grinding procedures demonstrated mean fracture strength values lower than the unprepared implants. When the mean values of the unprepared implants were compared with those of the prepared implants, the difference was found to be statistically significant. However, a deeper preparation did not further reduce the fracture strength. Several authors have investigated the effect of sandblasting, wet and dry grinding on the mechanical properties of Y-TZP ceramics. This influence on the flexural strength of ZrO_2 ceramics is contradictory and related to the volume percentage of transformed ZrO_2, which in turn depends on the metastability of the tetragonal to

TABLE 2 Multiple pairwise comparisons of the different subgroups using Wilcoxon rank sum test (significantly different when the value is $p < .05$)

Subgroup	*p* Value (adjusted)	Significance
D1 versus D2	1.0000	Not significant
D2 versus D3	1.0000	Not significant
D1 versus E1	1.0000	Not significant
D2 versus E2	1.0000	Not significant
E1 versus E2	1.0000	Not significant
E1 versus F1	0.0163	Significant
F1 versus F2	1.0000	Not significant
G1 versus G2	1.0000	Not significant
F1 versus G1	1.0000	Not significant
F2 versus G2	1.0000	Not significant

Test D1: uncoated Y-TZP-A BIO-HIP® implants (no exposure to the artificial mouth); test D2: uncoated Y-TZP-A BIO-HIP implants (exposure to the artificial mouth); test D3: uncoated Y-TZP-A BIO-HIP implants (exposure, no thermocycling); test E1: Y-TZP-A BIO-HIP ZiUnite® implants (no exposure + no preparation of the implant heads); test E2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + no preparation of the implant heads); test F1: Y-TZP-A BIO-HIP ZiUnite implants (no exposure + 0.5-mm chamfer); test F2: Y-TZP-A BIO-HIP ZiUnite implants (exposure + 0.5-mm chamfer); test G1: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 0.5-mm chamfer); test G2: Y-TZP-A BIO-HIP ZiUnite implants with crowns (exposure + 1-mm chamfer).

monoclinic phase transformation, the grinding severity, and the locally developed temperatures.[28–31] Severe grinding introduces deep surface flaws, which may become the strength determining if their length exceeds the depth of the grinding-induced surface compressive layer.[30–32] Either the flaws or the temperature changes introduced during the preparation of the abutment could have led to the aging of the material, resulting in lower fracture strength values.[33,34] The increase in monoclinic phase leads to a reduction in strength, toughness, and density, followed by micro-macrocracking and surface roughening.[35] In aqueous environments, this offers a path for the water to penetrate down into the specimen, creating corrosion effects on the Zr–O–Zr bonds.[27,36] The growth stage depends on several microstructural patterns: porosity, residual stresses, grain size, etc.[37] In the present study, the absence of water thermocycling during the exposure of the implants of subgroup D3 to the artificial mouth may have led to the higher mean fracture strength value of these implants in comparison with the implants of subgroup D2, which were exposed to the artificial mouth with water thermocycling. However, among the groups compared (D2 versus D3) no statistically significant difference was reported ($p > .05$).

Another point of discussion in the present study has been the difference in implant diameter among the compared groups, which was unavoidable because of the different manufacturing companies and could possibly have an impact on fracture resistance. Among the different groups, the ceramic implants had almost the same diameter (4.3 mm), which was higher than the diameter of Ti implants, resulting in an improved stress distribution around the implants' neck.[38] However, there was not an apparent influence of this parameter on the observed mean fracture strength values.

As far as the merits related to one-piece implant design is concerned, it should be mentioned that when applying one-piece implants for everyday practice, the surgery could be made flapless, with minimal surgical invasion and benefits in soft tissue preservation.[39,40] It has also been reported to be a safe procedure in terms of implant success, with percentages equivalent to healed site and delayed loading protocols.[41–43] Furthermore, the most common reported screw joint complications, consisting primarily of abutment screw loosening or screw fracture,[44–46] are avoided with one-piece implants. Other studies have also demonstrated that an intense inflammatory process, and thus significantly greater bone loss, was observed around two-piece implants when compared with one-piece implants, as the gaps, cavities, and hollow spaces, which have been described in two-piece implants, can be a trap for bacteria, even when a good marginal fit of implant components is present.[47–50] Another benefit of a one-piece implant design is that the implant can be inserted and immediately restored with a provisional crown. This may be of importance in cases of single-tooth replacement in the esthetic region.[51]

With regard to implantation, one disadvantage is that the implants have to be inserted into the perfect anatomical position, as only small corrections of the abutment's inclination are possible. Therefore, their initial positioning in the esthetic zone becomes even more critical because of the one-piece design.[39] Without the flexibility of an interchangeable abutment, these implants do not allow the use of any kind of attachments, and thus a conversion to overdentures. In case of implant head fracture, the removal of the integrated implant is unavoidable because there is no possible repair of the fractured implant.

Within the limits of this study, it may be concluded that the mean fracture strength of ZrO_2 implants ranged within the limits of clinical acceptance. However, some implants fractured at comparatively low loads, and some failed already during cyclic loading. The authors recognize that the ceramic implants do not show a Gaussian distribution as do metal implants, and implant fractures may occur at comparatively low loading forces, which poses a risk in clinical application. Furthermore, it may be concluded that the preparation of the implant heads had a significantly negative influence on the implant fracture strength. Although these early experimental data are encouraging, long-term clinical data are necessary before one-piece ZrO_2 implants may be universally recommended.

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