©Operative Dentistry, 2022, **47-4,** 461-472

Cement Choice and the Fatigue Performance of Monolithic Zirconia Restorations

LF Guilardi • GKR Pereira • JC Giordani • CJ Kleverlaan • LF Valandro • MP Rippe

Clinical Relevance

The clinical fatigue performance of cemented monolithic zirconia can be influenced by the cement choice. Proper selection of the cement system can enhance long-term fatigue performance.

SUMMARY

This study investigated the fatigue failure load of simplified monolithic yttria partially stabilized zirconia polycrystal restorations cemented to a dentin-like substrate using different luting systems. Disc-shaped ceramic (Zenostar T, 10 mm Ø × 0.7 mm thick) and dentin-like substrate (10 mm Ø × 2.8 mm thick) were produced and randomly allocated into eight groups, without or with thermocycling (TC=5-55°C/12,000×): "cement" (RelyX Luting 2 – glass ionomer cement [Ion], [Ion/TC]; RelyX U200 – self-adhesive resin cement [Self], [Self/TC]; Single Bond Universal+RelyX Ultimate – MDP-containing adhesive + resin cement [MDP-AD + RC], [MDP-AD + RC/ TC]; ED Primer II+Panavia F 2.0 – Primer + MDP-containing resin cement [PR + MDP-RC], [PR + MDP-RC/TC])). Each luting system was used as recommended by the manufacturer. Staircase methodology (20 Hz; 250,000 cycles) was applied for obtaining the fatigue failure loads. Fractographic characteristics were

Luis Felipe Guilardi, associate researcher, Post-Graduate Program in Oral Science (Prosthodontics Unit), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

Gabriel Kalil Rocha Pereira, MsciD, PhD, adjunct professor, Post-Graduate Program in Oral Science (Prosthodontics Units), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

João Carlos Giordani, undergraduate student, Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

Cornelis Johannes Kleverlaan, PhD, full professor, Department of Dental Materials Science, Academic Centre for Dentistry Amsterdam, Universiteit van Amsterdam and Vrije Universiteit, Amsterdam, Noord-Holland, The Netherlands

Luiz Felipe Valandro, MSciD, PhD, full professor, Post-Graduate Program in Oral Science (Prosthodontics Units), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

*Marilia Pivetta Rippe, MSciD, PhD, adjunct professor, Post-Graduate Program in Oral Science (Prosthodontics Units), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil

*Corresponding author: Avenue Roraima 1000, Building 26F, Room 2386, 97105-900, Santa Maria, Rio Grande do Sul, Brazil; e-mail: mariliarip@hotmail.com

http://doi.org/10.234/20-143-L

also assessed. At baseline, the Ion group presented the lowest fatigue load, although it was statistically similar to the Self group. The resin-based cement systems presented the highest fatigue performance, with the Ion group being only statistically equal to the Self group. Thermocycling influenced the groups differently. After aging, the MDP-AD + RC presented the highest mean, followed by the PR + MDP-RC and Self groups, while the Ion group had the lowest mean. Fractographic analysis depicted all failures as radial cracks starting at the zirconia intaglio surface. The luting system with MDP-containing adhesive applied prior to the resin cement presented the highest fatigue failure load after aging, presenting the best predictability of stable performance. Despite this, monolithic zirconia presents high load-bearing capability regardless of the luting agent.

INTRODUCTION

Monolithic zirconia restorations are an alternative to bilayer restorations in posterior teeth since they have not shown chipping or fractures after at least 68 months of clinical use,[1,2] and they allow less tooth reduction,[3,4] being used in reduced thickness (0.5-1.0 mm). Long-term clinical data on monolithic zirconia treatments are still scarce[5]; however, clinical studies with zirconia-based restorations have shown that fracture and retention loss are their main reasons for failure.[6-11]

When considering the factor "retention loss" for all-ceramic restorations, one of the aggravating characteristics is their internal relief. Unlike metal-ceramics, which have a certain primary friction to the dental substrate when cemented, all-ceramic crowns are not able to withstand such tension without damage.[12] According to Kelly,[12] this friction could induce tensile stress capable of generating internal cracks in the restoration, in addition to generating the radial tension effect (*Hoop stress*) caused by dental crowns' cylindrical shape when submitted to load (eg, luting procedure and chewing cycles). In this sense, the luting material plays an important role to compensate for this lack of primary friction and to prevent the restoration from debonding. Therefore, the choice of luting material should not be based on clinician preferences, but rather on scientific evidence that considers and compares specific protocols.

Several studies have demonstrated the benefits and importance of using techniques that not only promote a micromechanical bond, but also a strong, reliable and long-lasting chemical bond between tooth and ceramic restoration for greater longevity.[13-15] The quality of bonding interfaces is one of the major factors responsible for the fracture resistance of all-ceramic dental crowns since bulk fractures originate from defects on the restoration intaglio surface.[16] Also, these findings have been confirmed through fractographic analyses of clinically failed restorations[16] and using finite element predictions.[17] Therefore, it becomes clear that the use of different cement systems can potentially influence the retention and fatigue behavior of such restorations; factors which are largely related to the restoration longevity.[18]

The adhesive cementation should be performed whenever possible, as it generates higher bond and fatigue strength than conventional luting, which is related to a higher failure rate by retention loss.[11,19,20] Resin cements have better mechanical and optical properties, and higher resistance to abrasion and to hydrolysis,[21] despite their higher technical sensitivity and lower moisture tolerance. The introduction of functional monomers has improved the resin cement bond strength to zirconia,[13] but the effect of using different methacryloyloxy-decyl-dihydrogen-phosphate (MDP)-containing cement systems in the fatigue behavior of cemented monolithic zirconia is still unclear.

Moreover, the ability of luting systems to adequately fill the defects of the ceramic intaglio surface is another concern when cementing these restorations.[22] The fracture strength of ceramic materials is related to the size and number of defects present in their surface,[23] and unfilled defects can work as starting points for slow crack growth under constant masticatory stresses, and consequently cause early failure in the restoration.[24]

The hostile oral environment when associated to cyclic masticatory loads results in restoration failure due to long-term damage propagation, which constitutes fatigue failure.[25] This failure can be defined as ceramic fracture due to subcritical slow crack growth (SCG), which occurs under cyclic tensions lower than the normal strength of the material,[26] and SCG is accelerated in aqueous environments.[24] According to Ritter,[27] SCG can be explained as a chemical reaction that occurs between water and ceramics, by breaking their metal oxide bonds. In this way, the crack increases slowly, leading to strength decrease, and failure of restorations over time. Also, the cement's properties (eg, elastic modulus) may be affected by aging.[28]

From these standpoints, the aim of this study was to investigate the effect of using different luting systems on the fatigue performance of simplified monolithic zirconia specimens cemented to a dentin-like substrate, after applying stress by fatigue loading and thermal cycling. The null hypotheses tested were: 1) cement type and 2) aging will not affect the zirconia fatigue performance.

METHODS AND MATERIALS

To eliminate some production expenses (ie, CAD/CAM milling) and the complications associated with creating predictable contact between the piston and anatomically contoured ceramic while allowing the evaluation of the factors in this study, a simplified assembly (disc-shaped samples) for producing the restorative specimens was used. That approach is well validated in the literature[29-32] since it produces a stress distribution very similar to clinical scenarios where the stress concentration is higher at the luting interface on the intaglio ceramic surface, triggering the origin of failure.

Study Design

A second-generation yttria-stabilized tetragonal zirconia polycrystal (Y-TZP; 4.5% to 6.0% yttria content; Zenostar T; Wieland Dental, Ivoclar Vivadent; Schaan, Liechtenstein) indicated for framework and monolithic prosthetic restorations was used in the present study. The zirconia thickness used in the study was the minimal recommended by the manufacturer for monolithic posterior crowns, being 0.7 mm. The zirconia discs were cemented on flat dentin-like substrate discs (woven glass-fiber-filled epoxy resin; National Electrical Manufacturers Association [NEMA] grade G10, Accurate Plastics Inc, New York, USA; E_{G10}= 18.6 GPa - elastic modulus similar to wet dentin E_{dentin}= 18 GPa[29]). The final diameter of the specimens was 10 mm resembling the mean diameter of the occlusal surface of the first permanent molar.[33] The final thickness of the whole specimen set was 3.5 mm (G10 discs=2.8 mm, zirconia discs=0.7 mm), being equivalent to the mean thickness between the occlusal surface and the dental pulp chamber roof.[34,35]

Production of Specimens

Y-TZP Ceramic Discs—Zenostar T discs (98.5 mm Ø × 16 mm in thick) (n=200) were manually cut into small blocks (12 × 12 × 16 mm^3) with a diamond disc coupled to a handpiece attached to an electric motor (Perfecta LA 623T, 1000 at 40,000 rpm; W&H, Bürmoos, Austria). Next, metallic rings (Ø=12 mm) were glued to the parallel surfaces of the small blocks to guide the grinding in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, IL, USA) with #600 grit silicon carbide papers (SiC) and water-cooling to obtain zirconia cylinders with 12 mm of diameter.

Then, 0.94-mm thick slices were obtained by cutting under water-cooling with a diamond blade (Buehler-Series 15LC Diamond; Buehler) in a precision cutting machine (Isomet 1000, Buehler), resulting in 200 discs. The discs were manually polished on both sides with SiC papers (#600 and #1200 grit) to obtain a smooth surface, free from defects and with a final thickness of 0.86 mm. They were subsequently cleaned (ultrasonic bath with distilled water for 10 minutes) and dried, and then sintered in a specific furnace (heating rate of 600°C/h; temperature 1 of 900°C with a holding time of 0.5 h; heating rate of 200°C/h; and temperature 2 of 1450°C with a holding time of 2h; VITA Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Säckingen, Germany), followed by ultrasonic cleaning in 78% isopropyl alcohol for 5 minutes. The final dimensions of the zirconia discs were 10.0 mm in diameter and 0.7 (±0.02) mm in thickness.

Dentin-like Substrate Discs—NEMA G10 round rods (±250 mm length × 12.7 mm Ø) had their diameters reduced to 10 mm and then sliced in 3.0-mm thick discs (n=200) by the methodology previously described for the zirconia discs. After cutting, the discs were polished with SiC papers (#400 and #600 grit) until a final thickness of 2.8 mm, followed by ultrasonic cleaning in 78% isopropyl alcohol for 5 minutes.

Luting Procedure—Zirconia/Dentin-like Substrate

The intaglio surface of the all-zirconia discs was air-abraded for 10 seconds with aluminum oxide particles (Al_2O_3; 45 μm particle size) with oscillatory movements and a perpendicular angulation (90°) between the device tip and the specimen surface at a distance of 10 mm and at 2.8 bars of pressure. Next, the specimens were ultrasonically cleaned in distilled water for 5 minutes.

All the dentin-like substrate discs were etched with 10% hydrofluoric acid for 1 minute (HF etching), rinsed for 30 seconds, ultrasonically cleaned in distilled water for 5 minutes, and air-dried.

The specimens (zirconia and dentin-like substrate discs) were then randomly (www.randomizer.org) allocated into 8 groups (n=25) according to the study factors (cement and aging) (Table 1). The primers for each cement system were applied to the disc surfaces, and the cements were handled and applied following the manufacturers' instructions, as explained in Table 2.

After the primers were applied, each cement was mixed according to manufacturers' instructions (1:1 ratio) and applied on the dentin-like substrate disc. The zirconia discs were seated in their respective pairs under a uniform load of 250 g, which would be enough to produce a thin and uniform cement layer;[36] the cement excess was removed, and the cement was

Table 1: *Study Experimental Design*[a]

Cement Systems			Groups	
Classification	**Commercial Name (Brand), Elastic Modulus**	**General Composition**[b]	**Baseline**	**Aged (TC)**
Resin-modified glass ionomer cement (1-step)	RelyX Luting 2 (3M Oral Care) *E*=4 GPa[b]	Cement - Paste A: radiopaque FAS glass, proprietary reducing agent for self-cure, HEMA, water, opacifying agent; Paste B: methacrylated polycarboxylic acid, HEMA, water, potassium persulfate, non-reactive zirconia silica filler.	Ion	Ion/TC
Self-adhesive resin cement (1-step)	RelyX™ U200 (3M Oral Care) *E*=6.6 GPa[b]	Cement - Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	Self	Self/TC
Self-etching primers + MDP-containing adhesive resin cement (2-steps)	ED Primer II + Panavia F 2.0 (Kuraray Noritake) *E*=18.3 GPa[c]	Cement - Paste A: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica dl-Camphorquinone, catalysts; Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments. Primers - Liquid A: HEMA, 10-MDP, N-methacryloyl-5-aminosalicylic acid, water, accelerators. Liquid B: N-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators	PR + MDP-RC	PR + MDP-RC/TC
MDP-containing universal adhesive + adhesive resin cement (2-steps)	Single Bond Universal + 3M RelyX Ultimate (3M Oral Care) *E*=7.7 GPa[b]	Cement - Base paste: methacrylate monomers, radiopaque, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, dark cure activator for Scotchbond Universal Adhesive (3M Oral Care). Adhesive - MDP, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	MDP-AD + RC	MDP-AD + RC/TC

Abbreviations: FAS, fluoroaluminosilicate; HEMA, 2-hydroxyethylmethacrylate; MDP, methacryloyloxydecyl-dihydrogen-phosphate; TC, thermocycling; AD, adhesive; RC, resin cement.

[a] *Cement Systems: classification, commercial name, brand and elastic modulus, and general composition; aging: baseline and aged (Thermocycling - TC: 12,000 cycles between 5 ºC and 55 ºC, 30 seconds dwell time; 4 seconds transfer time); and group codes.*

[b] *Manufacturer's data.*

[c] *Li and others*[60]

Table 2: *Luting Procedures for the Different Cement Systems*

Cement	Groups	Surface Treatment	
		Epoxy Resin	Zirconia
Resin-modified glass ionomer cement (1-step); RelyX Luting 2	Ion and Ion/TC	After HF etching (see the luting procedure section), a silane-coupling agent[29] (RelyX Ceramic Primer; 3M Oral Care) was applied for 5 s, and gently air-dried.	After air-abrasion and cleaning (see the luting procedure section), the specimens were vigorously air-dried.
Self-adhesive resin cement (1-step); RelyX U200	Self and Self/TC		
Self-etching primers + MDP-containing adhesive resin cement (2-steps); ED Primer II + Panavia F 2.0	PR + MDP-RC and PR + MDP-RC/TC	After HF etching (see the luting procedure section), the Panavia system ED Primers II, liquids A and B, were mixed (ratio 1:1) and applied on the surface, the mixture was left to react for 30 s and primer excess was removed by gentle air-drying for 5 s.	After air-abrasion and cleaning (see the luting procedure section), the specimens were vigorously air-dried.
MDP-containing universal adhesive + adhesive resin cement (2-steps); Single Bond Universal + RelyX Ultimate	MDP-AD + RC and MDP-AD + RC/TC	After HF etching (see the luting procedure section), the Single Bond Universal Adhesive (3M Oral Care) was applied and left to react for 20 s and the excess was removed by gentle air-drying for 5 s.	After air-abrasion and cleaning (see the luting procedure section), the specimens were vigorously air-dried. Single Bond Universal Adhesive (3M Oral Care) was applied and left to react for 20 s and the excess was removed by gentle air-drying for 5 s.

Abbreviations: Ion, glass ionomer cement; Self, self-adhesive resin cement; MDP-AD + RC, MDP-containing adhesive + resin cement; PR + MDP-RC, Primer + MDP-containing resin cement; TC, thermocycling; MDP, methacryloyloxydecyl-dihydrogen-phosphate.

light-cured (1200 mW/cm^2, 440-480 nm, Radii-cal, SDI; Bayswater, Australia) for 20 seconds through the occlusal ceramic surface and for 20 seconds on each side (0°, 90°, 180° and 270°) of the specimen set.

Artificial aging - Thermocycling 'TC'

Half of the specimens from each cement system underwent 12,000 thermal cycles between two water baths, 5°C and 55°C (30 seconds dwell time and 4 seconds transfer time (model 521-6D, Ethik Technology, Vargem Grande Paulista, Brazil), 1 day after cementation and stored for four days after thermocycling. After cementation, the specimens not thermocycled were stored in distilled water at 37°C in a laboratory oven (Laboratory Thermo incubator, Model 502, FANEM, São Paulo, Brazil) for four days.

Fatigue Failure Load Testing - Staircase Method

The specimens for each group were numbered and randomized (www.randomizer.org) to determine their test sequence. The fatigue tests were executed in an electric machine (Instron ElectroPuls E3000, Instron Corp, Norwood, MA, USA) over a flat steel base and through the Staircase sensitivity method.[37] The cyclic loads (250,000 pulse cycles; 20 Hz frequency; wet testing) were applied to the center of the disc surface on the zirconia side by a 40-mm Ø hemispheric stainless-steel piston (Figure 1).[29,38] The fatigue test parameters (initial load = ~60% of the mean of load-to-failure tests; and step-size = ~5% of the initial load) were obtained from the mean of the static load-to-failure tests (0.5 mm/min crosshead speed [EMIC DL 2000, São José dos Pinhais, Brazil] of 5 specimens until the specimen's failure, ie, auditory perception of cracking by a single trained operator). This procedure was performed for each group. An adhesive tape (110 μm thick) was placed on the zirconia surface to improve stress spreading during load application,[39,40] and a polyethylene sheet (10 μm thick) was placed between the piston and the cemented set to reduce contact stress concentration,[41] both in order to avoid contact damage (Hertzian's cone cracks).

For the fatigue tests, the first specimen of each group was tested with the initial load (~60% of the mean of the

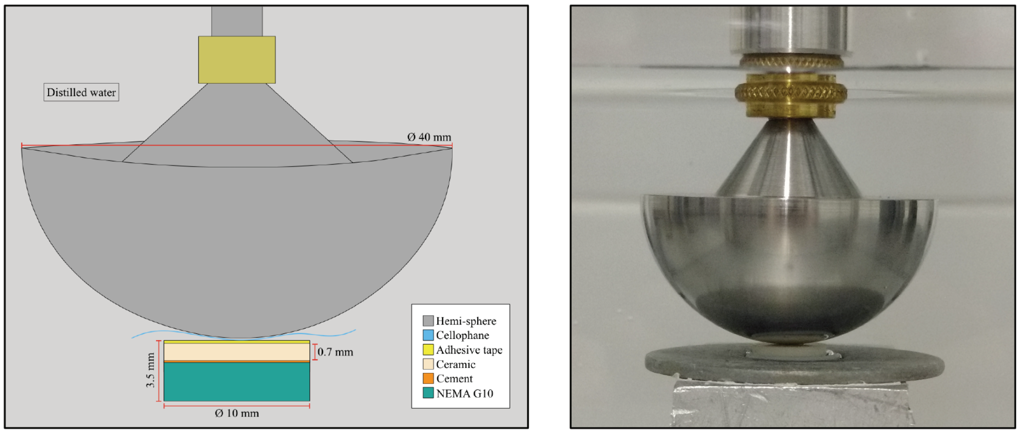

Figure 1. *Fatigue test assembly - schematic drawing of the set and photograph of the hemispheric stainless-steel piston (40 mm Ø) used to apply the load in the center of the specimens' occlusal surface, submerged in distilled water.*

load-to-failure test), and then one step-size (~5% of initial load) was added or subtracted for the next specimen depending on the previous specimen's survival (+1 step) or failure (-1 step) to the predefined cycles (250,000). The test was sequentially performed until a minimum of 15 specimens were tested after the up-and-down method had started, being, according to Collins,[37] enough to achieve an accurate fatigue measurement.

Fractographic Analysis

After fatigue testing, the fractured specimens were evaluated in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss, Göttingen, Germany) to determine the crack location. The crack was marked to be cut perpendicularly in two halves in a high-precision diamond saw (Isomet 1000, Buehler). Representative specimens were selected for scanning electron microscopy analysis (Secondary Electron Detector [SE], VEGA3 Tescan, Brno-Kohoutovice, Czech Republic) to better describe their failure characteristics. The images were taken to analyze the radial crack in two different perspectives; a typical fractographic analysis of a debonded zirconia specimen after fracture (350× and 1000× magnification), and an analysis of a transversal view of the crack (250× of magnification), as mentioned above for the specimens that remained bonded after fracture.

Statistical Analysis

Two-way analysis of variance (ANOVA, IBM SPSS Statistics Program v24 for Windows, IBM Corp, New York, NY, USA; α=0.05) was used to determine the influence of the independent variables (cement and thermocycling) and their interaction (cement + thermocycling) on the dependent variable (fatigue failure load).

The mean load for fatigue failure (L_f), standard deviation (SD), and 95% confidence interval (CI) were calculated using the Dixon and Mood method,[42] which involves the maximum-likelihood estimation (overlapping confidence intervals) and assumes a normal distribution of the data,[37] as described in previous studies.[43,44]

RESULTS

Based on two-way ANOVA, a statistically significant influence was observed for the cement (p<0.001) and aging factors (p<0.001), and their interaction (cement + aging; p<0.001) on the fatigue failure load data.

The mean monotonic load-to-failure values, the parameters for fatigue tests and results, and the graphics of fatigue survival/failure patterns for each group are described in Table 3 and Figure 2.

Considering the baseline condition, the Ion cement group had the lowest fatigue load (1530.00), being statistically equal to the Self group (1570.00). After aging, the MDP-AD + RC/TC presented the highest fatigue values (1957.50), while the Ion/TC (1551.67) presented the lowest ones. Aging had no deleterious effect on fatigue loads (Table 3).

Radial crack was the fracture pattern observed for all groups and it originated from the intaglio ceramic surface. Figure 3 shows the fractographic characteristics under two perspectives; in a specimen in which the zirconia fragments separated after failure (Figures 3A and 3B), and in a transversal cut of a sectioned specimen that remained cemented after failure (Figure 3C). No cone-cracks were observed.

DISCUSSION

The present study demonstrated that the luting protocol affects the monolithic zirconia fatigue failure load, refuting the first null hypothesis, and that the aging process applied was not enough to jeopardize the mechanical behavior of the restorative assembly,

Table 3: *Mean of Monotonic Load-to-Failure Test (n=5)*[a]

Groups	Mean Monotonic Load-to-Failure (n)	Initial Load for Fatigue Test (n	Step-size Increment (n)	Mean Load for Fatigue Failure L_f (SD)	95% CI[b]	Load Decrease (%)
Ion	1998.55	1200	60	1530.00 (286.32)	1319.23 - 1740.77 B	23
Ion/TC	1773.75	1060	50	1551.67 (40.60)	1518.21 - 1585.13 c	13
Self	2382.53	1430	70	1570.00 (294.89)	1369.02 - 1770.98 AB	34
Self/TC	2181.63	1310	65	1754.17 (122.98)	1661.67 - 1846.67 b	20
PR + MDP-RC	2160.00	1300	65	1847.86 (119.10)	1764.04 - 1931.68 A	14
PR + MDP-RC/TC	2124.57	1275	65	1767.14 (58.93)	1723.05 - 1811.23 b	17
MDP-AD + RC	2172.17	1300	65	1820.00 (55.70)	1763.77 - 1876.23 A*	16
MDP-AD + RC/TC	2237.54	1340	65	1957.50 (64.48)	1905.91 - 2009.09 a*	13

Abbreviations: Ion, glass ionomer cement; Self, self-adhesive resin cement; MDP-AD + RC, MDP-containing adhesive + resin cement; PR + MDP-RC, Primer + MDP-containing resin cement; TC, thermocycling; MDP, methacryloyloxydecyl-dihydrogen-phosphate

[a] *Fatigue test parameters: initial load for fatigue tests (~60% of mean monotonic load-to-failure), step-size (~5% of initial load). Fatigue results: mean load for fatigue failure (L_f)(SD) and 95% confidence interval (CI); and percentage of decreasing load comparing the mean value of monotonic load-to-failure and the mean load for fatigue failure.*

[b] *Statistical analysis for fatigue test - Dixon & Mood statistical method[42] (confidence intervals overlapping): different uppercase letters represent statistically significant difference for different cement systems on baseline; different lowercase letters mean statistical difference for different cement systems after thermocycling; and asterisk (*) represents statistically significant difference between baseline and aged between the same cement system.*

accepting the second null hypothesis. The study results showed that bonding the air-abraded monolithic zirconia using an MDP-containing universal adhesive plus an adhesive resin cement (MDP-AD + RC system) provided the best long-term fatigue failure load results. Also, the investigated zirconia ceramic (Zenostar T) can endure high fatigue loads, even in a thin (0.7 mm) thickness, thus providing a conservative dental option for monolithic crowns in the posterior region of the mouth, being able to withstand even the highest biting forces during nocturnal bruxism, which can reach 800 N.[45]

In a recent systematic review and meta-analysis, Thammajaruk and others[46] concluded that mechanical and chemical pre-treatments are determinant on the bond strength to zirconia, particularly when MDP-containing primers are used, both with and without aging. Kern[47] reviewed and compared the best available clinical and laboratory evidence for successful bonding of dental oxide ceramic restorations and concluded that the association of air-abrasion at a moderate pressure (0.1-0.25 MPa) with the use of primers and/or resin cements containing a phosphate monomer (MDP) provides long-term durable bonding to zirconia ceramic. In the present study, better results after aging were achieved when luting the monolithic zirconia using an MDP-containing adhesive (RelyX Ultimate system).

Luting the zirconia ceramic with the resin-modified glass ionomer cement (RelyX Luting 2) led to the worst fatigue behavior after aging. The lower fatigue performance of crowns cemented with glass-ionomer systems has been shown previously.[48,49] This improvement provided by resin cements is related to their greater ability to create a strong adhesion between a dentin-like substrate and zirconia.[19] Furthermore, resin-based cements have a higher modulus of elasticity and flexural strength than ionomer-based cements, enabling a better foundation.[50]

The *in vitro* studies should simulate the aging of the materials and of the adhesive interface[18] since the restorations are exposed to different challenges in the mouth (ie, humidity, variations in temperature and pH).[51] The aging can degrade the adhesive bonding through some factors, such as cement stiffness reduction,[28] hydrolytic degradation of the materials' polymer matrix by water penetration, and fatigue of the adhesive interface due to the mismatch of linear thermal expansion coefficients (different rates of shrinkage and expansion) between bonded materials during temperature changes,[52] thereby affecting long-term success of the restoration.[18] According to Lu and

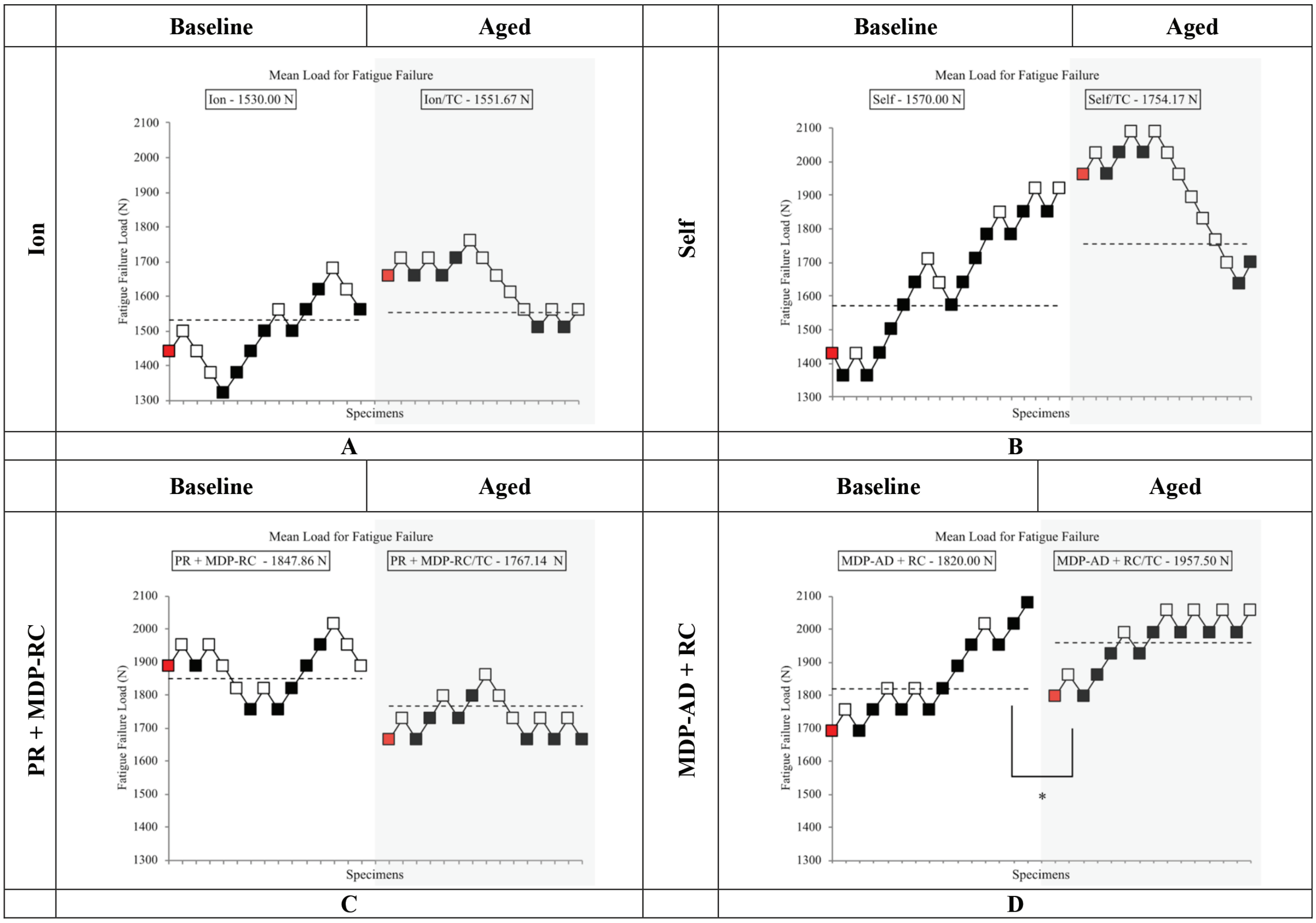

Figure 2. *Staircase survival and failure patterns for each group (n=20 or a minimum of 15 specimens tested after the up-and-down characters have been started; red marks). The horizontal dashed lines represent the mean load for fatigue failure of each group; black marks mean intact specimens and white marks mean failed ones. Asterisk (*) indicates a statistically significant difference between baseline and aged groups for the same cement system based on Table 3.*

others,[53] aging in water can degrade the bond strength and stiffness (ie, decrease of the elastic modulus) of cement agents, leading to stress redistribution in the ceramic crown, reducing its load-bearing capacity.

In the present study, we did not observe a negative effect of thermocycling on the assembly fatigue behavior, even following the number of cycles recommended by Andreatta and others[52] as being deleterious for bond strength values between ceramic material and resin cement. However, Zhao and others[54] reported that slow thermal cycling (3000 thermal cycles - 5°C/55°C, where each thermal cycle took 15 minutes) is more effective than a fast-changing temperature profile to promote the aging process of the bonding interface in materials with low thermal diffusivity (eg, zirconia). In this sense, the transfer time used in our study (only 4 seconds) could explain why thermal cycling did not deleteriously impact the fatigue load. In a previous study,[36] the aging process did affect the zirconia fatigue behavior when the zirconia was air-abraded with aluminum oxide (45 μm particle) and bonded with the Panavia F2.0 system (Kuraray, Noritake, Ukayama, Japan). That could be explained since the aging process was more aggressive in that study,[36] which applied 60 days of storage in distilled water at 37°C additionally to the thermocycling protocol (12,000 thermal cycles 5°C-55°C), which may have allowed water to penetrate and degrade the bonding interface.[52] Hygroscopic expansion is material dependent and sometimes it can exceed the amount of polymerization shrinkage, overcompensating it and leading to internal expansion stress, endangering the restoration integrity.[55]

Indeed, the aging significantly increased the fatigue failure load of the MDP-AD + RC system, and according to de Oyagüe and others,[56] this could be explained by the long carbonyl chain of the acidic functional monomer

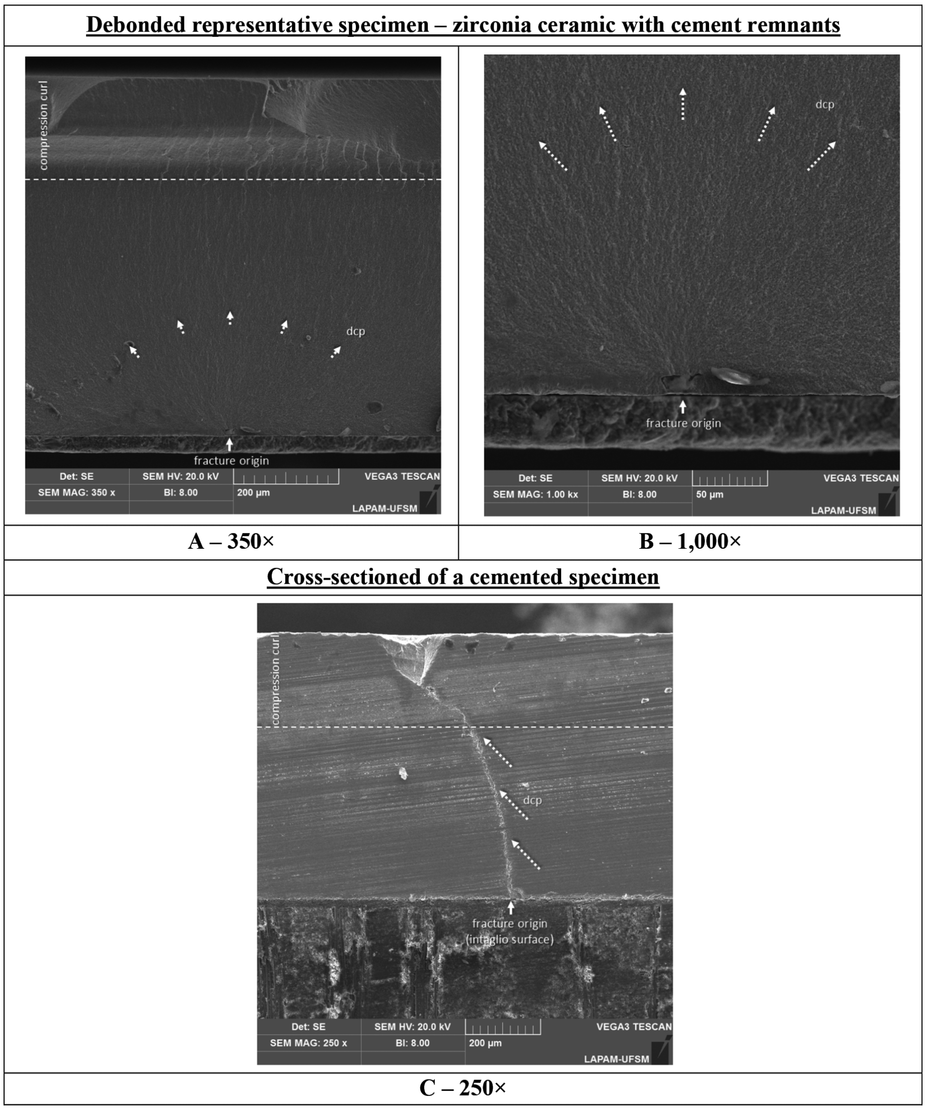

Figure 3. *SEM images showing the typical fractographic characteristics after fatigue failure under two perspectives: A and B show a specimen where ceramic fragments were separated after failure; C shows a transverse cut of a sectioned specimen that remained cemented after failure (radial crack). White arrows point to the site of fracture origin; dashed horizontal line indicates the compression curl; white dashed arrows indicate the direction of crack propagation (dcp).*[59]

present in the MDP formulation that is relatively stable to hydrolysis. From Zhao and others,[54] the presence of the methacrylate-modified polyalkenoic acid has a moisture-stabilizing effect on the Single Bond Universal Adhesive, which can explain its better behavior when subjected to aging. Furthermore, only the MDP-AD + RC system received application of an adhesive on the zirconia surface, and as adhesives have lower viscosity, they are able to better wet and fill in the ceramic surface irregularities, improving the bond strength and reducing the water penetration at the interface.[57] Still, when restorative materials absorb water, their dimensions and structural integrity may be affected, and in this case the shrinkage stress of the adhesive may be partially relieved by the water uptake, neutralizing the tensions at the adhesive interface, and better distributing the stress during loading,[55] consequently increasing the fatigue failure load of the restorative set-up.

As stated by Zhang and others,[25] a post-failure fractographic analysis can provide valuable guidance to find the fracture origin and other failure characteristics. In our study, we only found radial cracks originating from the ceramic intaglio surface (Figure 3), and no surface contact damage (Hertzian's cone cracks) was found.[38] Such findings are very important since radial cracks in the monolithic ceramic crowns can propagate and result in bulk fracture, one of the most common failure modes of all-ceramic restorations.[29]

The present study applied a simplified and standardized model, which eliminated some testing variables, and isolated the factors under study. It also followed some recommendations such as wet testing and the use of a hemispheric piston with a minimal diameter (40 mm) to create clinically sized contacts of 0.5 to 3 mm diameter (clinical wear facet size) at pressures of 5 to 890 MPa when applying realistic average maximum biting forces (100 to 700 N).[38] However, besides using a dentin-like substrate as a substitute for the human dentin, a uniaxial load was applied without clinical sliding contact, so the mechanical conditions of the oral environment were only partially reproduced.[26] Furthermore, in an attempt to reduce time spent on the fatigue testing and without significant impact on the zirconia fatigue behavior,[43] the load frequency applied (20 Hz) was much higher than a normal chewing frequency (0.94 - 2.17 Hz[58]).

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. The fatigue failure load of monolithic zirconia cemented to a dentin-like substrate was influenced by the luting system.
2. Aging had no damaging effect on the fatigue failure load of the monolithic zirconia specimens.
3. The use of an MDP-containing adhesive associated with a resin cement promoted better fatigue results after aging.

Acknowledgments

This work is part of the fulfillment for the requirements of the PhD degree (LFG) in the Post Graduate Program in Oral Sciences at the Faculty of Dentistry, Federal University of Santa Maria-RS, Brazil. This study was financed in part by the Brazilian Federal Agency for Coordination of Improvement of Higher Education Personnel - CAPES (Finance Code 001), and in part by the scholarship financial support at Academisch Centrum Tandheelkunde Amsterdam (ACTA) CAPES/NUFFIC program: NUFFIC Dutch Organization for Internationalization in Education; Project # 056/14; Process # 88881.145663/2017-01). The authors are grateful to Ivoclar Vivadent for providing their materials free of charge.

Conflicts of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any kind in any product, service, and/or company that is presented in this article.

(Accepted 21 October 2021)

REFERENCES

1. Bömicke W, Rammelsberg P, Stober T, & Schmitter M (2017) Short-term prospective clinical evaluation of monolithic and partially veneered zirconia single crowns *Journal of Esthetic and Restorative Dentistry* **29(1)** 22-30. DOI: 10.1111/jerd.12270
2. Moscovitch M (2015) Consecutive case series of monolithic and minimally veneered zirconia restorations on teeth and implants: Up to 68 months *International Journal of Periodontics and Restorative Dentistry* **35(3)** 315-323. doi: 10.11607/prd.2270
3. Edelhoff D & Sorensen JA (2002). Tooth structure removal associated with various preparation designs for anterior teeth *Journal of Prosthetic Dentistry* **87(5)** 503-509. doi: 10.1067/mpr.2002.124094
4. Edelhoff D & Sorensen JA (2002). Tooth structure removal associated with various preparation designs for posterior teeth *International Journal of Periodontics and Restorative Dentistry* **22(3)** 241-249.
5. Spitznagel FA, Boldt J, & Gierthmuehlen PC (2018) CAD/CAM ceramic restorative materials for natural teeth *Journal of Dental Research* **97(10)** 1082-1091. doi: 10.1177/0022034518779759
6. Sailer I, Fehér A, Filser F, Gauckler LJ, Lüthy H, & Hämmerle CHF (2007) Five-year clinical results of zirconia frameworks for posterior fixed partial dentures *International Journal of Prosthodontics* **20(4)** 383-388.
7. Tinschert J. Schulze KA, Natt G, Latzke P, Heussen N, & Spiekermann H (2008) Clinical behavior of zirconia-based fixed partial dentures made of DC-Zirkon: 3-year results *International Journal of Prosthodontics* **21(3)** 217-222.
8. Schmitter M, Mussotter K, Rammelsberg P, Stober T, Ohlmann B, & Gabbert O (2009) Clinical performance of extended zirconia frameworks for fixed dental prostheses: Two-year results *Journal of Oral Rehabilitation* **36(8)** 610-615. Doi: 10.1111/j.1365-2842.2009.01969.x
9. Wolfart S, Harder S, Eschbach S, Lehmann F, & Kern M (2009) Four-year clinical results of fixed dental prostheses with zirconia substructures (Cercon): End abutments vs. cantilever design *European Journal of Oral Sciences* **117(6)** 741-749.
10. Roediger M, Gersdorff N, Huels A, & Rinke S (2010) Prospective evaluation of zirconia posterior fixed partial dentures: Four-year clinical results *International Journal of Prosthodontics* **23(2)** 141-148.
11. Rinke S, Gersdorff N, Lange K, & Roediger M (2013) Prospective evaluation of zirconia posterior fixed partial dentures: 7-year clinical results *International Journal of Prosthodontics* **26(2)** 164-171. doi: 10.11607/ijp.3229
12. Kelly JR (2016) *Ceramics in dentistry: principles and practice*. **1st edition** Hanover Park, IL: Quintessence Publishing Co, Inc.
13. Inokoshi M, De Munck J, Minakuchi S, & Van Meerbeek B (2014) Meta-analysis of bonding effectiveness to zirconia ceramics *Journal of Dental Research* **93(4)** 329-334. doi: 10.1177/0022034514524228
14. Luthra R & Kaur P (2016) An insight into current concepts and techniques in resin bonding to high strength ceramics *Australian Dental Journal* **61(2)** 163-173. doi: 10.1111/adj.12365
15. Özcan M & Bernasconi M (2015) Adhesion to zirconia used

for dental restorations: A systematic review and meta-analysis *Journal of Adhesive Dentistry* **17(1)** 7-26. doi: 10.3290/j.jad.a33525

16. Kelly JR, Giordano R, Pober R, & Cima MJ (1990) Fracture surface analysis of dental ceramics: Clinically failed restorations *International Journal of Prosthodontics* **3(5)** 430-440.

17. De Jager N, de Kler M, & van der Zel JM (2006) The influence of different core material on the FEA-determined stress distribution in dental crowns *Dental Materials* **22(3)** 234-242. doi: 10.1016/j.dental.2005.04.034

18. Arola D (2017) Fatigue testing of biomaterials and their interfaces *Dental Materials* **33(4)** 367-381. doi: 10.1016/j.dental.2017.01.012

19. Rippe MP, Amaral R, Oliveira FS, Cesar PF, Scotti R, Valandro LF, & Bottino MA (2015) Evaluation of tensile retention of Y-TZP crowns cemented on resin composite cores: Effect of the cement and Y-TZP surface conditioning *Operative Dentistry* **40(1)** E1-E10. doi: 10.2341/13-310-L

20. Campos F, Valandro LF, Feitosa SA, Kleverlaan CJ, Feilzer AJ, de Jager N, & Bottino MA (2017) Adhesive cementation promotes higher fatigue resistance to zirconia crowns *Operative Dentistry* **42(2)** 215-224. doi: 10.2341/16-002-L

21. Stawarczyk B, Keul C, Eichberger M, Figge D, Edelhoff D, & Lümkemann N (2017) Three generations of zirconia: From veneered to monolithic Part II *Quintessence International* **48(6)** 441-450. doi: 10.3290/j.qi.a38157

22. Thompson JY, Rapp MM, & Parker AJ (1998) Microscopic and energy dipersive x-ray analysis of surface adaptation of dental cements to dental ceramic surfaces *Journal of Prosthetic Dentistry* **79(4)** 378-383. doi: 10.1016/s0022-3913(98)70148-9

23. Griffith AA (1921) The phenomena of rupture and flow in solids Philosophical Transactions of the Royal Society **221** 163-198.

24. Gonzaga CC, Cesar PF, Miranda WG Jr, & Yoshimura HN (2011) Slow crack growth and reliability of dental ceramics *Dental Materials* **27(4)** 394-406. doi: 10.1016/j.dental.2010.10.025

25. Zhang Y, Sailer I, & Lawn BR (2013) Fatigue of dental ceramics *Journal of Dentistry* **41(12)** 1135-1147. doi: 10.1016/j.jdent.2013.10.007

26. Wiskott HW, Nicholls JI, & Belser UC (1995) Stress fatigue: Basic principles and prosthodontic implications *International Journal of Prosthodontics* **8(2)** 105-116.

27. Ritter JE (1995) Predicting lifetimes of materials and material structures *Dental Materials* **11(2)** 142-146. doi: 10.1016/0109-5641(95)80050-6

28. Lu C, Wang R, Mao S, Arola D, & Zhang D (2013) Reduction of load-bearing capacity of all-ceramic crowns due to cement aging *Journal of the Mechanical Behavior of Biomedical Materials* **17** 56-65. doi: 10.1016/j.jmbbm.2012.08.003

29. Kelly JR, Rungruanganunt P, Hunter B, & Vailati F (2010) Development of a clinically validated bulk failure test for ceramic crowns *Journal of Prosthetic Dentistry* **104(4)** 228-238. doi: 10.1016/S0022-3913(10)60129-1

30. de Kok P, Pereira GKR, Fraga S, de Jager N, Venturini AB, & Kleverlaan CJ (2017) The effect of internal roughness and bonding on the fracture resistance and structural reliability of lithium disilicate ceramic *Dental Materials* **33(12)** 1416-1425. doi: 10.1016/j.dental.2017.09.018

31. Monteiro JB, Riquieri H, Prochnow C, Guilardi LF, Pereira GKR, Borges ALS, de Melo RM, & Valandro LF (2018) Fatigue failure load of two resin-bonded zirconia-reinforced lithium silicate glass-ceramics: Effect of ceramic thickness *Dental Materials* **34(6)** 891-900. doi: 10.1016/j.dental.2018.03.004

32. Pereira GKR, Graunke P, Maroli A, Zucuni CP, Prochnow C, Valandro LF, Caldas RA, & Bacchi A (2019) Lithium disilicate glass-ceramic vs translucent zirconia polycrystals bonded to distinct substrates: Fatigue failure load, number of cycles for failure, survival rates, and stress distribution *Journal of the Mechanical Behavior of Biomedical Materials* **91** 122-130. doi: 10.1016/j.jmbbm.2018.12.010

33. Ferrario VF, Sforza C, Tartaglia GM, Colombo A, & Serrao G (1999) Size and shape of the human first permanent molar: A Fourier analysis of the occlusal and equatorial outlines *American Journal of Physical Anthropology* **108(3)** 281-294. doi: 10.1002/(SICI)1096-8644(199903)108:3<281::AID-AJPA4>3.0.CO;2-#

34. Harris EF & Hicks JD (1998) A radiographic assessment of enamel thickness in human maxillary incisors *Archives of Oral Biology* **43(10)** 825-831. doi: 10.1016/s0003-9969(98)00061-2

35. Sulieman M, Addy M, & Rees JS (2005) Surface and intra-pulpal temperature rises during tooth bleaching: An *in vitro* study *British Dental Journal* **199(1)** 37-40. doi: 10.1038/sj.bdj.4812558

36. Guilardi LF, Pereira GKR, Giordani JC, Kleverlaan CJ, Valandro LF, & Rippe MP (2019) Effect of zirconia surface treatment, resin cement and aging on the load-bearing capacity under fatigue of thin simplified full-contour Y-TZP restorations *Journal of the Mechanical Behavior of Biomedical Materials* **97** 21-29. doi: 10.1016/j.jmbbm.2019.04.050

37. Collins JA (1993) *Failure of Materials in Mechanical Design: Analysis, Prediction, Prevention*. **2nd edition**. New York: John Wiley & Sons.

38. Kelly JR (1999) Clinically relevant approach to failure testing of all-ceramic restorations *Journal of Prosthetic Dentistry* **81(6)** 652-661. doi: 10.1016/s0022-3913(99)70103-4

39. Quinn GD, Ives LK, & Jahanmir S (2005) On the nature of machining cracks in ground ceramics: Part I: SRBSN strengths and fractographic analysis *Machining Science and Technology* **9(3)** 169-210.

40. Wachtman JR Jr, Capps W, & Mandel J (1972) Biaxial flexure tests of ceramic substrates *Journal of Materials* **7(2)** 188-194.

41. ISO 6872:2015. Dentistry - Ceramic materials (2015) *International Organization for Standardization* **4ed.** Switzerland: Geneva.

42. Dixon WJ & Mood AM (1948) A method for obtaining and analyzing sensitivity data *Journal of the American Statistical Association* **43(241)** 109-126. doi: 10.1080/01621459.1948.10483254

43. Fraga S, Pereira GKR, Freitas M, Kleverlaan CJ, Valandro LF, & May LG (2016) Loading frequencies up to 20Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic *Journal of the Mechanical Behavior of Biomedical Materials* **61** 79-86. doi: 10.1016/j.jmbbm.2016.01.008

44. Pereira GKR, Silvestri T, Amaral M, Rippe MP, Kleverlaan CJ, & Valandro LF (2016) Fatigue limit of polycrystalline zirconium oxide ceramics: Effect of grinding and low-temperature aging

Journal of the Mechanical Behavior of Biomedical Materials **61** 45-54. doi: 10.1016/j.jmbbm.2016.01.006

45. Nishigawa K, Bando E, & Nakano M (2001) Quantitative study of bite force during sleep associated bruxism *Journal of Oral Rehabilitation* **28(5)** 485-491. doi: 10.1046/j.1365-2842.2001.00692.x

46. Thammajaruk P, Inokoshi M, Chong S, & Guazzato M (2018) Bonding of composite cements to zirconia: A systematic review and meta-analysis of *in vitro* studies *Journal of the Mechanical Behavior of Biomedical Materials* **80** 258-268. doi: 10.1016/j.jmbbm.2018.02.008

47. Kern M (2015) Bonding to oxide ceramics—laboratory testing versus clinical outcome *Dental Materials* **31(1)** 8-14. doi: 10.1016/j.dental.2014.06.007

48. Anami LC, Lima JMC, Valandro LF, Kleverlaan CJ, Feilzer AJ, & Bottino MA (2016) Fatigue resistance of Y-TZP/porcelain crowns is not influenced by the conditioning of the intaglio surface *Operative Dentistry* **41(1)** E1-12. doi: 10.2341/14-166-L

49. Lawson NC, Jurado CA, Huang C-T, Morris GP, Burgess JO, Liu P-R, Kinderknecht KE, Lin CP, & Givan DA (2019) Effect of surface treatment and cement on fracture load of traditional zirconia (3Y), translucent zirconia (5Y), and lithium disilicate crowns *Journal of Prosthodontics* **28(6)** 659-665. doi: 10.1111/jopr.13088

50. Saskalauskaite E, Tam LE, & McComb D (2008) Flexural strength, elastic modulus, and pH profile of self-etch resin luting cements *Journal of Prosthodontics* **17(4)** 262-268. doi: 10.1111/j.1532-849X.2007.00278.x

51. Gale MS & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27(2)** 89-99. doi: 10.1016/s0300-5712(98)00037-2

52. Andreatta Filho OD, Junho de Araújo MA, Bottino MA, Nishioka RS, & Menezes MM (2005) Study of thermocycling effect on the bond strength between an aluminous ceramic and a resin cement *Journal of Applied Oral Science* **13(1)** 53-57. doi: 10.1590/s1678-77572005000100011

53. Lu C, Wang R, Mao S, Arola D, & Zhang D (2013) Reduction of load-bearing capacity of all-ceramic crowns due to cement aging *Journal of the Mechanical Behavior of Biomedical Materials* **17** 56-65. doi: 10.1016/j.jmbbm.2012.08.003

54. Zhao L, Jian Y-T, Wang X-D, & Zhao K (2016) Bond strength of primer/cement systems to zirconia subjected to artificial aging *Journal of Prosthetic Dentistry* **116(5)** 790-796. doi: 10.1016/j.prosdent.2016.03.020

55. Bociong K, Szczesio A, Sokolowski K, Domarecka M, Sokolowski J, Krasowski M, & Lukomska-Szymanska M (2017) The influence of water sorption of dental light-cured composites on shrinkage stress *Materials (Basel)* **10(10)** 1142. doi: 10.3390/ma10101142

56. de Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic *Dental Materials* **25(2)** 172-179. doi: 10.1016/j.dental.2008.05.012

57. de Souza G, Hennig D, Aggarwal A, & Tam LE (2014) The use of MDP-based materials for bonding to zirconia *Journal of Prosthetic Dentistry* **112(4)** 895-902. doi: 10.1016/j.prosdent.2014.01.016

58. Po JM, Kieser JA, Gallo LM, Tésenyi AJ, Herbison P, & Farella M (2011) Time-frequency analysis of chewing activity in the natural environment *Journal of Dental Research* **90(10)** 1206-1210. doi: 10.1177/0022034511416669

59. Scherrer SS, Lohbauer U, Della Bona A, Vichi A, Tholey MJ, Kelly JR, van Noort R, & Cesar PF (2017) ADM guidance-ceramics: Guidance to the use of fractography in failure analysis of brittle materials *Dental Materials* **33(6)** 599-620. doi: 10.1016/j.dental.2017.03.004

60. Li L-L, Wang Z-Y, Bai Z-C, Mao Y, Gao B, Xin H-T, Zhou B, Zhang Y, & Liu B (2006) Three-dimensional finite element analysis of weakened roots restored with different cements in combination with titanium alloy posts *Chinese Medical Journal (Engl)* **119(4)** 305-311.