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## Fatigue behavior of ultrafine tabletop ceramic restorations



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### ARTICLE INFO

#### Article history:

Received 8 November 2017

Received in revised form

29 May 2018

Accepted 7 June 2018

#### Keywords:

Ceramics

Mechanical stress

Fixed dental prosthesis

Fatigue test

### ABSTRACT

**Objective.** The goal of this study was to investigate the fatigue life, failure modes, and stress distribution of partial ultrafine restorations for posterior teeth in different ceramics.

**Methods.** Sixty standard tabletop preparations in epoxy resin G10 received lithium-silicate-based zirconia-reinforced (ZLS) or hybrid ceramic (PIC) restorations in 0.5- or 1-mm thickness bonded with resin cement. The same cycling protocol was applied for all specimens, which consisted of 5000 cycles at 200 N, followed by 450-N cycles until the specimens' fracture or the suspension of the test after  $1.5 \times 10^6$  cycles. Axial load was carried out with a 4 Hz frequency in Biocycle V2 equipment (Biopdi, São Carlos, SP), with samples immersed in water. The presence of cracks and/or fractures was checked every  $2.5 \times 10^5$  cycles, and the survival analysis was performed with the number of cycles in which each specimen failed. All specimens were evaluated by stereomicroscopy and scanning electron microscopy (SEM). After data tabulation, Kaplan–Meier and Mantel–Cox (log-rank test) analyses were performed, followed by multiple pairwise comparison, all with a significance level of 5%, and Weibull analysis. Through three-dimensional finite element analysis, stress distribution and maximum principal stresses in the posterior occlusal veneers were evaluated by comparison of different types of substrate (G10, enamel/dentin, enamel), thicknesses, and ceramic materials.

**Results.** Zirconium-reinforced lithium silicate restorations with 0.5-mm thickness (ZLS.5) showed lower fatigue strength compared with that of 1.0-mm hybrid ceramic restorations (PIC1), and both were similar to other restorations (PIC.5 and ZLS1) (log-rank test,  $\chi^2 = 11.2$ ;  $df = 3$ ;  $p = 0.0107 < 0.05$ ). ZLS groups presented random defects that culminated in fracture, whereas PIC groups presented defects that increased with mechanical fatigue after some cycling time. Stereomicroscope images show radial cracks due to the translucency of the material. There was no damage caused by the applicator. MPS (maximum principal stress) distributions were similar for the different substrate types, but the highest modulus of elasticity showed slightly lower stress concentration.

**Significance.** PIC is more likely to be used in thinner thickness than indicated by the manufacturer, with fatigue strength similar to that of thicker ZLS restorations.

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<https://doi.org/10.1016/j.dental.2018.06.017>

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## 1. Introduction

Preservation of tooth structure has always been a challenge for restorative dentistry [1]. For centuries, various techniques and materials have been developed to restore and reproduce dental structures [2]. Ceramics are vitreous materials, therefore friable, and thus, to play a role similar to that of a dental structure, must support masticatory forces without degrading [3]. The microstructure of ceramics significantly affects their behavior, so chemical composition, mechanical properties, laboratory processing type, thickness, and cementation method must be considered when the material type is selected. New minimally invasive restorative clinical approaches from a biomimetic perspective were facilitated by the evolution of metal-free ceramics and the advent of adhesive cementation [1,4], with materials that resemble natural tissues used for the repair of lost dental structures [5]. For the treatment of severe erosive lesions, ultrafine restorations adhesively cemented have been shown to be a conservative alternative to traditional onlays or total crowns in posterior teeth [6,7].

Various new types of ceramics, reinforced with more resistant materials like zirconia, have been developed, such as Vita Suprinity (ZLS, zirconia-reinforced lithium silicate), which has an elastic modulus closer to that of enamel, 65 GPa [8], and others with softer, polymer-like materials in their composition. Vita Enamic, for example (PIC, 14% organic polymer-infiltrated ceramic), is a hybrid material that combines ceramic and composite characteristics [9,10], presenting a low modulus of elasticity, closer to that of dentin, around 30 GPa [8]. Both materials can be found in CAD/CAM blocks and are indicated for partial posterior tooth restoration.

Good results in fracture resistance have been shown with the use of indirect monolithic ceramic restorations [11,12], which can be applied for partial preparation in posterior teeth, even in thin thicknesses [13], and are a good choice for worn dentition, requiring minimally invasive rehabilitation, restoring lost structures with minimal or no dental preparation [14,15].

Fatigue tests have been presented as an efficient method for the evaluation of the mechanical properties of long-term dental materials [3,16–18]. This type of test promotes the occurrence of chemical phenomena like slow crack growth, mechanical process degradation such as “hydraulic pumping”, and internal friction in the walls of microcracks that occur in ceramic materials [18–21].

The purpose of this *in vitro* study was to evaluate the fatigue life of ultrafine, 0.5 and 1 mm, tabletop restorations for posterior teeth, in two different ceramics, a lithium silicate zirconia-reinforced (ZLS) and a hybrid ceramic (PIC), both adhesively cemented in a dentin-like material (G10).

## 2. Materials and methods

### 2.1. Specimen preparation

Sixty standard tabletop preparations with a simplified occlusal reduction corresponding to a lower second molar were milled in G10 fiber glass-reinforced epoxy resin (Protec, São Paulo,

Brazil), a dentin-analogous material [22]. Specimens were embedded in acrylic resin cylinders (TDV, Pomerode, Brazil) 2 mm below the cement-enamel junction, and one master preparation was scanned (inLab SW4.2, SironaDental Systems GmbH, Bensheim, Germany). The information was sent to Cerec 3 software (v3.03, SironaDental Systems GmbH). Restorations were waxed in two occlusal thicknesses, 0.5 mm or 1.0 mm, and were also scanned. Vita Zahnfabrik blocks (Bad Säckingen, Germany) were milled to obtain restorations in Vita Suprinity with 0.5-mm (ZLS.5) or 1-mm thickness (ZLS1) or in Vita Enamic with 0.5-mm (PIC.5) or 1-mm thickness (PIC1). Restorations were cleaned in an ultrasonic bath with isopropyl alcohol for 10 min and dried. Following the manufacturer's instructions, milled Suprinity restorations underwent crystallization firing in a Vita Vacumat 6000 MP furnace (Vita Zahnfabrik). Polishing procedures were carried out with the respective commercial polishing kits (Vita Suprinity polishing set and Vita Enamic polishing set, both Vita Zahnfabrik). G10 preparations were randomly chosen and cleaned with 10% hydrofluoric acid (Condac porcelana, FGM Produtos Odontológicos Ltda, Joinville, Brazil) for 60 s, rinsed with air/water spray, and dried with oil-free air jets, after which a layer of the A + B adhesive system (Kuraray Noritake, Okayama, Japan) was applied by microbrush for 30 s, simulating a dental procedure. ZLS and PIC restorations were also cleaned in an ultrasonic bath and dried, after which the Clearfil Ceramic Primer (Kuraray Noritake) was applied for 60 s and light-cured. Panavia F 2.0 (Kuraray Noritake) resin cement was applied to internal restoration surfaces then seated in position with a 750-g apparatus; excess cement was removed with a microbrush, an air barrier was applied to cover the margins, and light polymerization was performed with Bluephase Style (Ivoclar Vivadent, Schaan, Liechtenstein) for 20 s on each surface. Cementation margins were finished and polished. All specimens were kept in 37 °C distilled water for 30 h prior to being tested.

### 2.2. Mechanical testing and reliability analysis

All specimens were subjected to the same cycling protocol. Axial loading was carried out at a frequency of 4 Hz in Biocycle V2 equipment (Biopdi, São Carlos, Brazil), with samples immersed in water. Specimens received an initial load of 200 N for 5000 cycles, followed by 450 N until failure, through a stainless-steel sphere with a 4.6-mm-diameter indenter centered with three-point contacts. Testing was limited to a maximum of  $1.5 \times 10^6$  cycles and was interrupted every  $2.5 \times 10^5$  cycles to check for the presence of cracks and/or fractures with the aid of adequate illumination and a stereomicroscope. An acetate strip was positioned between the applicator and the ceramic surface. The number of cycles until failure was recorded and used for survival analysis by Kaplan–Meier and Mantel–Cox tests (log-rank test), followed by multiple pairwise comparisons, all with a 5% significance level (GraphPad Prism version 7, La Jolla, CA, USA).

Failure probability was calculated for each time interval: 0–250,000, 250,000–500,000, 500,000–750,000, 750,000–1,000,000, 1,000,000–1,500,000, and 1,500,000–2,000,000 cycles (90% of bilateral confidence interval – Synthesis 9, Weibull ++ 9, Reliasoft). 2 parameter

Weibull failure probability analysis provided the beta ( $\beta$ ) value, which is a shape parameter and describes the behavior of the failure rate over time.  $\beta < 1$  indicates that the failure rate decreased over time, while a failure rate of  $\beta \sim 1$  indicates that there was no variation over time, and  $\beta > 1$  means that the failure rate increased over time [23,24]. The eta parameter ( $\eta$ ) represents the characteristic life of specimens, in which 63.2% of failures occurred [25].

### 2.3. Fractographic analysis

All samples were first inspected by light-polarized stereomicroscopy (Discovery V20 Zeiss, Jena, Germany) for identification of fracture marks and probable failure origin. When necessary, samples were gold-sputtered (Emitech SC7620 Sputter Coater, East Sussex, UK) and analyzed by scanning electron microscopy (SEM; XL 20, FEI Company, Czech Republic).

### 2.4. Finite element analysis (FEA)

FEA was performed for the evaluation of stress distribution in posterior occlusal veneers, comparing different substrate types (G10, enamel, or enamel/dentin), restoration thicknesses (0.5 or 1.0 mm), and ceramic types (ZLS or PIC).

### 2.5. Modeling

3D models were obtained by the scanning of G10 preparations by means of an inEos Blue scanner (Sirona Dental Systems) and replication of the proposed preparations. STL (stereolithography) file formats were transferred to CAD software, Rhinoceros 5.0 (McNeel North America, Seattle, WA,

**Table 1 – Materials' mechanical properties.**

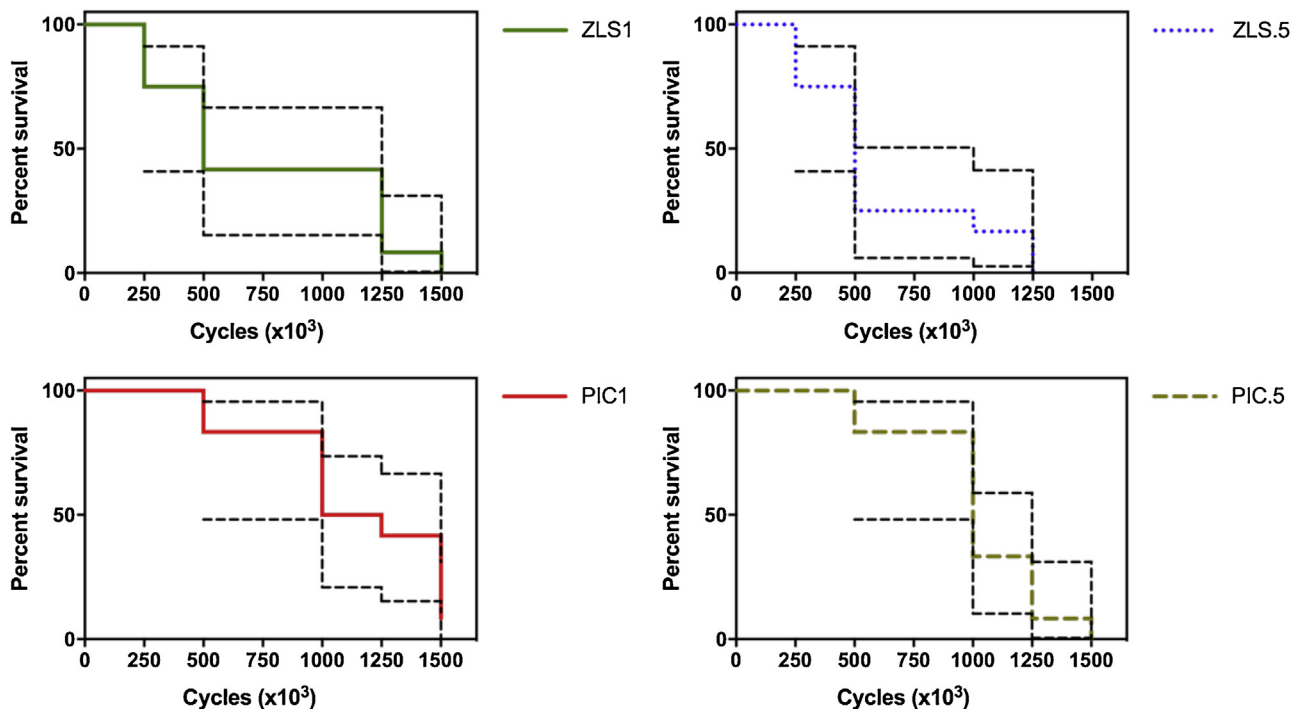
Material	Elastic moduli [GPa]	Poisson's coefficient	Reference
ZLS	65.6	0.23	Ramos et al. [8]
PIC	34.7	0.28	Ramos et al. [8]
Panavia F	3	0.35	Yi and Kelly [27]
G10	14.9	0.31	Kelly et al. [22]
Enamel	84.1	0.33	Zarone et al. [28]
Dentin	18.6	0.32	Zarone et al. [28]
Structural steel	200	0.30	Ansys library
Acrylic resin	26	0.38	Ansys library

USA), according to the BioCAD protocol [26] and then they were imported into Ansys software (version 17.0, Ansys, Canonsburg, PA, USA) for the preprocessing phase of FEA. The mechanical properties of the materials were identified based on the literature (Table 1). Interfaces were considered perfect-bonded, and the cement layer was 50  $\mu\text{m}$  thick. Solids were considered isotropic, linear, and homogeneous. The contact areas between sphere and a laboratory specimen were measured using a microscope and replicated in the virtual models. The 450-N load was applied perpendicular to the surfaces distributed at three occlusal contacts. Maximum principal stress (MPS) was the criterion analyzed.

## 3. Results

### 3.1. Fatigue testing

Kaplan–Meier test survival curves are shown in Fig. 1. The comparison between the experimental groups by the Mantel–Cox statistic detected a statistically significant difference between the analyzed conditions (log-rank test,  $\chi^2 = 11.2$ ,



**Fig. 1 – Survival graphs of experimental groups according to the number of cycles until failure ( $\times 10^3$ ). Dotted lines represent the upper and lower limits of their 95% confidence intervals.**

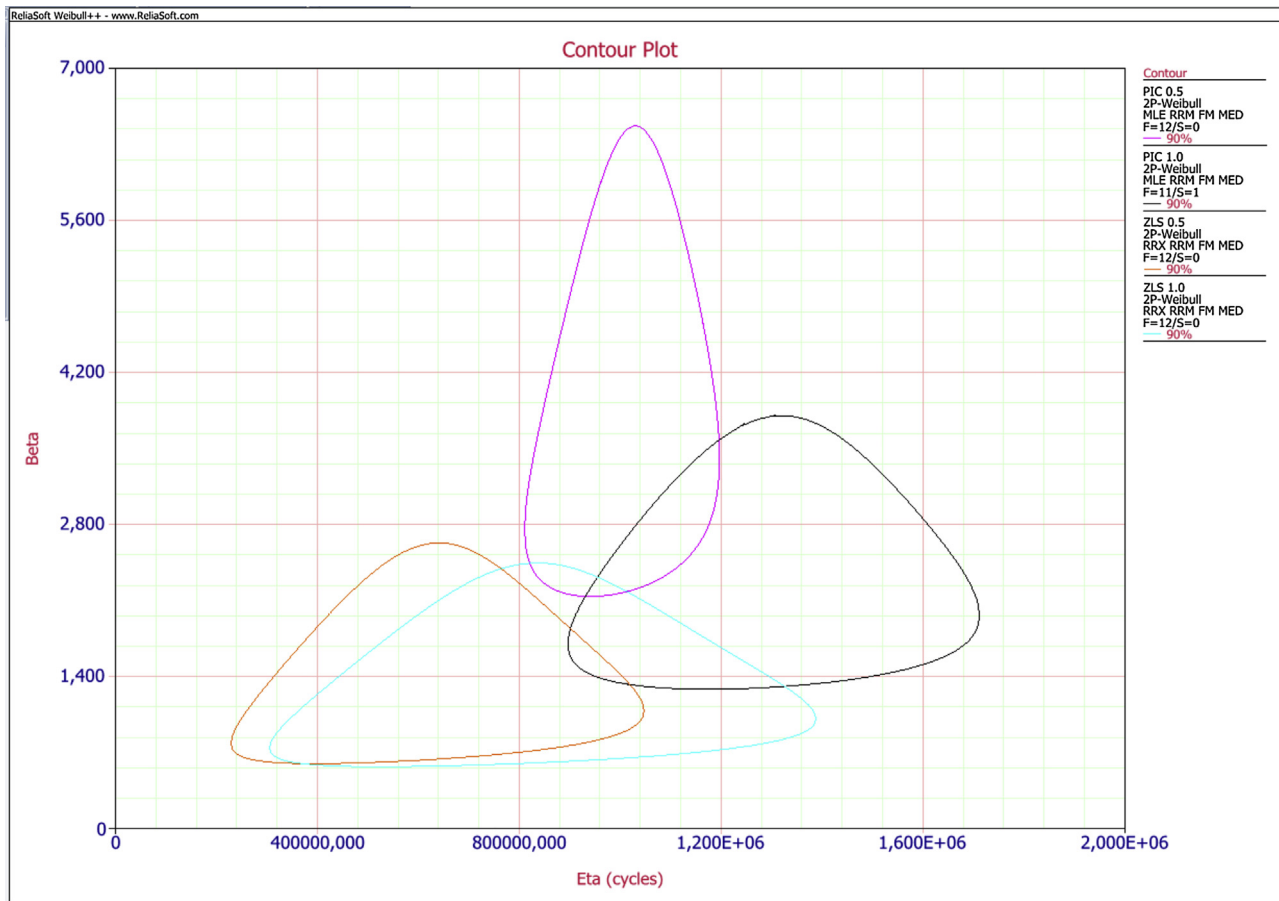
**Table 2 – Kaplan Meier statistic of the number of cycles until failure and homogeneous groups of the experimental groups tested in the present study.**

	Number of cycles (x10 <sup>3</sup> )						Homogeneous groups
	250	500	750	1000	1250	1500	
ZLS.5	0.750	0.250	–	0.167	0.000	–	B
ZLS1	0.750	0.417	–	–	0.083	0.000	AB
PIC.5	–	0.833	–	0.333	0.083	0.000	AB
PIC1	–	0.833	–	0.500	0.417	0.083	A

Missing values were not calculated due to lack of data.

**Table 3 – Weibull distribution parameters,  $\beta$  and  $\eta$ , and respective lower and upper limits for the 90% confidence interval of the experimental groups.**

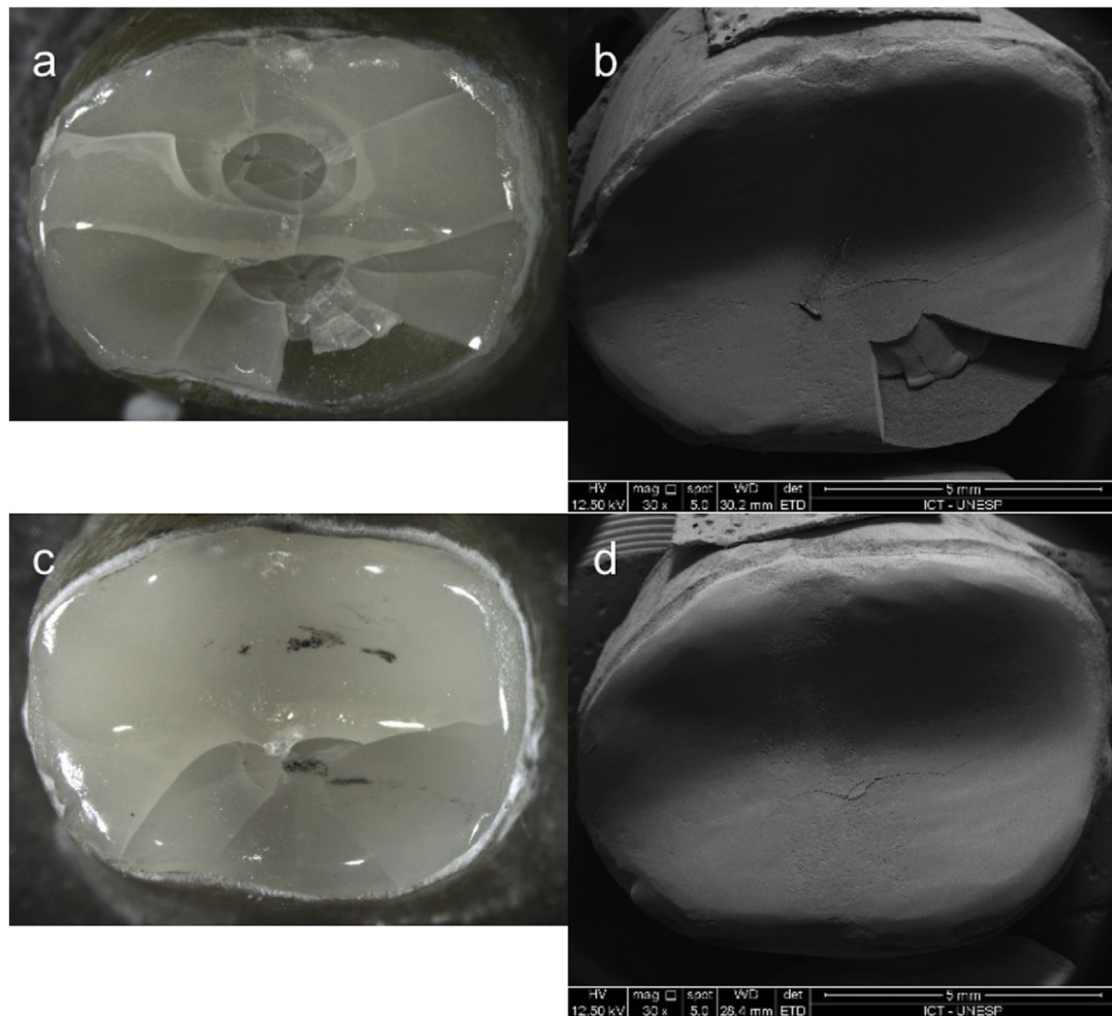
	Beta ( $\beta$ )	Lower/upper limits	Eta ( $\eta$ )	Lower/upper limits
PIC.5	3.9	2.5–5.9	990652.9	867883.3–1,1E + 06
PIC1	2.4	1.6–3.6	1,2E + 06	994351–1,5E + 06
ZLS.5	0.9	0.5–1.5	377834.5	220707.9–646822.5
ZLS1	0.9	0.6–1.6	487690	288685.2–823881.6



**Fig. 2 – Contour plot of the confidence intervals of the experimental groups.**

$df = 3, p = 0.0107 < 0.05$ ; Table 2). Considering the opposite survival rates, we observed that ZLS.5, with 1,250,000 cycles, had a rate of 0, while PIC1 had a rate of 0.417 (Table 2, Fig. 1). Thus, the possibility of a specimen of the PIC1 group surviving  $1.25 \times 10^6$  cycles at 450N without failure is 41.7%, while the probability of a specimen of the ZLS.5 group surviving the same conditions is 0%. Survival rates decreased with

increased numbers of cycles. Pairwise comparison showed that zirconium-reinforced lithium silicate ceramic restorations with 0.5-mm thickness (ZLS.5) had lower fatigue strength compared with 1.0-mm-thick hybrid ceramics (PIC1). The other restorations (PIC.5 and ZLS1) resembled all experimental groups.



**Fig. 3 – Stereomicroscope and SEM images. (a and b) ZLS.5; (c and d) ZLS1.**

ZLS samples presented random defects that culminated in fracture ( $\beta \sim 1$ ), whereas PIC samples presented defects that increased with the mechanical fatigue after some cycling ( $\beta > 1$ ) (Table 3). Although PIC samples were statistically similar due to the overlap of the confidence intervals (Fig. 2), PIC.5 presented the highest  $\beta$  value, indicating a lower variability of the results, that is, the fracture of the crowns almost always occurred between  $7.5 \times 10^5$  and  $1.0 \times 10^6$  cycles, with PIC generally taking a larger number of cycles before failure onset. The mean times for failure in years, based on the number of cycles the specimens survived and calculated by the software Synthesis 9, Weibull ++ 9, Reliasoft for the tested groups, were 102 (PIC.5), 124 (PIC1), 45 (ZLS.5), and 56 (ZLS1).

Only one sample from the ZLS group presented an adhesive failure between restoration and substrate. Overall, PIC fractures resulted in minor damage and fewer fragments compared with ZLS fractures. Stereomicroscope images showed radial and cone cracks due to the translucency of the material (Figs. 3 and 4). Micrographs of the same specimens (Figs. 3 and 4, right column) showed that fracture occurred in the volume of the material, and the damage on the occlusal surface was minimal or nonexistent.

### 3.2. FEA

Stress distribution patterns were similar for all analyzed models, and the areas with the highest stress concentration were found below the load application points. Increasing the thickness of restorations reduced the stress concentrations at both cementation and occlusal surfaces. ZLS presented higher concentrations of stresses in the adhesive interface when compared with PIC. The substrate with the highest modulus of elasticity had a slightly lower stress concentration in ceramic restoration (Fig. 5).

## 4. Discussion

This in vitro study evaluated the fatigue life of ultrafine table-top restorations for posterior teeth in both ZLS and PIC hybrid ceramics. Although manufacturers recommend 1.5 mm minimum thickness for partial posterior restorations, sometimes it is necessary to use less material, either to preserve healthy dental structure or to restore areas with limited inter-occlusal space. The present study found that 1-mm hybrid ceramics

performed significantly better in the fatigue test compared with 0.5-mm ZLS. This may be associated with the fact that the microstructure of PIC contains polymer-filled pores, and this combination of materials aims to increase fracture resistance as well as resistance to flexion [29–33].

Elastic modulus and fracture resistance are factors that must be taken into account in the selection of materials, since they are directly related to the materials' behavior against loads. ZLS, because of its higher elastic modulus, has shown a higher stress concentration at the ceramic adhesive interface in finite element analysis [34,35]. It appears that a ceramic with a lower elastic modulus, closer to that of dentin, can better withstand loads and better contain crack propagation [8,29,36,37], better distributing tensions [38]. In fact, PIC performed better at higher thickness (1 mm) than did ZLS at lower thickness (0.5 mm), surviving a larger number of cycles, with a statistically significant difference between the analyzed conditions and the other crowns, PIC.5 and ZLS1, which resembled all experimental groups. It can be seen in the finite element analysis that the increase in the thickness of the restorations significantly reduced the concentrations of stresses on internal and external surfaces, that is, luting and the occlusal interface, respectively. Decreasing the thickness of a restoration to less than 1 mm has a significant impact on increasing

its susceptibility to failure and therefore interfering in its clinical performance [35].

The substrate with a higher modulus of elasticity presented a slightly lower stress concentration [35], demonstrating that the larger the elastic modulus of the substrate, the greater the load required for fracture.

The susceptibility of material degradation to slow crack growth is another factor that interferes in long-term ceramic behavior and must be taken into account as being directly related to its microstructure. PIC presents a stress corrosion coefficient (n-value) almost three times higher than that of ZLS and is therefore much less susceptible to slow cracking [8]. PIC took longer to fail than did ZLS (Tables 1 and 3).

Differences were observed during the preparation of the restorations. The machining time for PIC was faster than that for ZLS. Edge chipping was also noted when restorations were finished after CAD/CAM processing. Thinner ZLS presented more irregularities than thicker ZLS and PIC at both thicknesses, suggesting better machinability. The machinability of glass-ceramic materials is directly related to their friability index [39], not so much to their hardness or resistance to fracture [40]. Due to their lower friability, polymer-infiltrated ceramics show good machinability and better finishing [29,31,36,41,42].

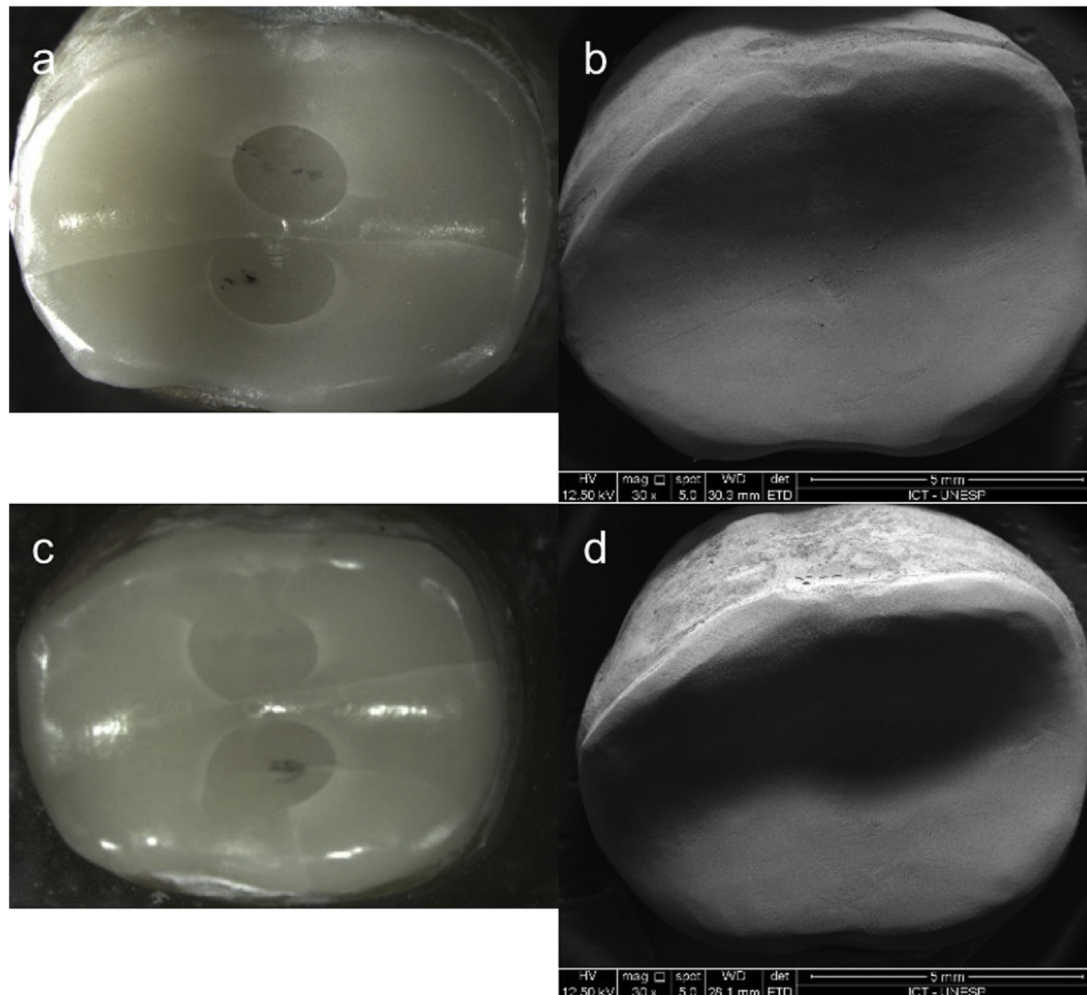
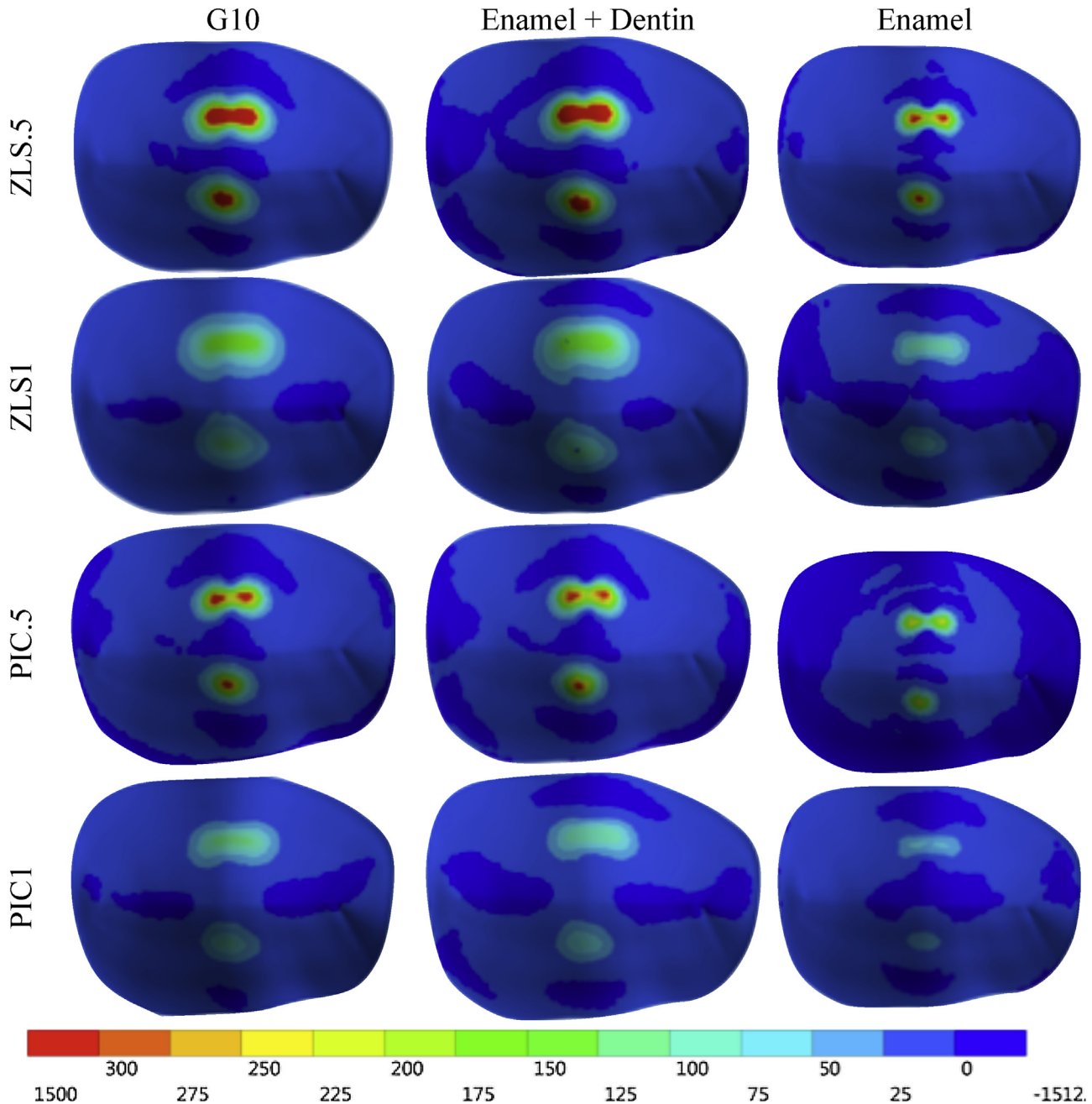


Fig. 4 – Stereomicroscope and SEM images. (a and b) PIC.5; (c and d) PIC1.



**Fig. 5 – Maximum principal stresses (MPS) for the models studied comparing G10, enamel + dentin and enamel substrates. Standardized scale.**

Through our analysis of the average life to failure (Weibull ++), we can say that, while the data do not necessarily reflect what happens in vivo, since there are other variables in the mouth, they are an attempt to estimate, under test conditions, how many years a restoration of each type of material would survive. However, the mean time to failure calculated for all groups was longer than the clinical trial times, although clinical trials are still necessary to evaluate restoration longevity.

The fatigue protocol used in the present study was considered efficient, since it resulted in failures similar to those found clinically, originating in the cementing surface rather than caused by the indenter [34,38,43,44]. Initially,

defects were classified as “cone cracks” and “radial cracks” by stereomicroscope analysis, then discarded in SEM analysis. Occlusal surfaces analyzed by micrographs showed that specimens had little or no damage, and that the origin of the defects occurred on the inner restoration surface. Thus, the apparent stereomicroscope cracks occurred inside the material and were visualized due to its transparency, and many of them did not reach the occlusal surfaces of the restorations.

The role of adhesion in the success of direct or indirect inlays, onlays, veneers, or crown restorations is critical; the final resistance of the tooth/restoration complex is dependent on this bond stability [6,7,35,45–48]. In the present study,

only one sample from the ZLS group showed adhesion failure between the restoration and the substrate after the fatigue test, so adhesion was considered efficient and did not affect the performance of the samples tested.

Tabletop restorations, 0.5 mm thick in this study, are considered mechanically favorable because they receive predominantly axial compressive forces [13]. This fact was confirmed by finite element analysis, which verified that the areas with the greatest concentrations of stress were detected, in all cases, just below the points of load application, in the interface, away from the edges of the restorations. This finding is in agreement with that from another work, which also reported that most fractures originated on the cementation surface, where the greatest tensile forces occurred [49].

G10 was chosen as the substrate in this study because it is a dentin-like material, with similar elastic modulus and adhesiveness [22], thus reducing the standardization bias of the samples in relation to the variability that could occur in natural teeth. The type of substrate is also related to the fracture resistance of restorative materials, a dependence even more pronounced in fine and ultrafine restorations [35,38]. Finite element analysis confirmed that the G10 behavior was similar to that of the simulated natural enamel/dentin substrate in the stress distributions in all models evaluated. However, the substrate with the highest modulus of elasticity, composed only of enamel, presented a lower stress concentration, distributing loads more uniformly in the volume of the restorations, due to its lower deformation capacity. With regard to the elastic modulus of the substrate, it has been verified that cement thickness can also interfere in restoration longevity: the thicker the cement, the smaller the elastic modulus, and the earlier the chance of restoration failure [35].

## 5. Conclusions

Within the limitations of this study and based on the results, it can be concluded that:

- (1) thin zirconium-reinforced lithium silicate ceramic restorations (ZLS.5) presented lower fatigue strength when compared with 1.0-mm-thick hybrid ceramic restorations (PIC1); and
- (2) the concentration of ZLS stresses at the adhesive interface was higher when compared with that for PIC.

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