UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

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Residual stress within the porcelain veneer of 3-unit zirconia fixed dental prostheses with different framework designs measured by nanoindentation

Estresse residual na porcelana de revestimento de próteses parciais fixas de 3 elementos com diferentes desenhos de infra-estrutura mensurado por nanoendentação

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Tese constituída por artigos apresentada a Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Doutor em Ciências no programa de Ciências Odontológicas Aplicadas, área de concentração Reabilitação Oral.

Orientador: Prof. Dr. Gerson Bonfante

Versão Corrigida

BAURU

2017

Pavesi Fardin, Vinicius
F222a Residual stress within the porcelain veneer of 3-unit zirconia fixed dental prostheses with different framework designs measured by nanoindentation / Vinicius Pavesi Fardin. – Bauru, 2017.
100 p.: il. ; 31cm

Tese (Doutorado) – Faculdade de Odontologia de Bauru. Universidade de São Paulo

Orientador: Prof. Dr. Gerson Bonfante

Nota: A versão original desta tese encontra-se disponível no Serviço de Biblioteca e Documentação da Faculdade de Odontologia de Bauru – FOB/USP.

FOLHA DE APROVAÇÃO

ACKNOWLEDGEMENTS

In gratitude I thank God for enlightening and guiding my life.

I am grateful to the University of São Paulo – Bauru School of Dentistry, Bauru, SP, Brazil and specially grateful to its Department of Prosthodontics and Periodontology for their special assistance during the last six years, which contribuited a lot for my development.

I would like to thank my special supervisor Professor Gerson Bonfante. A human being that was always motivated to share his experiences and concepts with others. Also, he introduced me to his son, Assistant Professor Estevam Bonfante, one of the brightest faculties of the Bauru College of Dentistry. His support was so important for achievement of all stages. I deeply admire his professional and family life.

My appreciation to my supervisor Associate Professor Paulo Guilherme Coelho. A brilliant researcher who gave to me the opportunity to improve my knowledge with the select group of students and researchers from the Department of Biomaterials and

Biomimetics of New York University, NY, USA in 2016. His carrer shows exactly how competent and proactive professional he is.

I would like to thank Assistant Professor Ronaldo Hirata, Associate Professor Rodrigo Neiva, Dr. Lucasz Witek, Dr. Nick Tovar and Mr. Dindo. I really appreciated the attention gave to me when I was in trouble.

A special thanks goes to the dear friends from the New York University: Daniel Navarro, João Barbosa, Heloísa Marão, José Carlos Romanini Junior, Dimorvan Bordin, Arthur Castellano, Eduardo Cáceres, Pablo Atria, Rodrigo from Venezuela, Michelle Bowers, Lindiane Rigo, Camila Sampaio, Marla Cuppini and Helora Moura. The days have become much easier with their presence.

I also thank the special professors of the Post-Graduation Bauru College of Dentistry: Luiz Fernando Pegoraro, Accácio Lins do Valle and Ana Lúcia Pompéia Fraga de Almeida. They were always able to support us at the postgraduation dental offices.

Many thanks to my Bauru roommates: Lucas Cambiaghi and Henrique Quevedo. Also, thanks to all the colleagues from Post-Graduation Bauru College of Dentistry, in special to Patrick Alves,

Thereza Pacheco, Ilana Ramalho, Adolfo Coelho and Vitor Guarçoni.

Special thanks to the friends of mine met in the beginning of the Post-Graduation Bauru College of Dentistry. This true friendship, for at least 5 years, have resulted in the best moments lived in Bauru. I could say that André Porporatti, Denise Gusmão, Aline Baía, Letícia Korb, Yuri Martins, Adriana Braga, Dylton Augusto Neto and Karen Ayub were present in this period of my life.

I cannot forget the friends from the city that I was born. Thanks so much for the effort to celebrate some special days with me. In particular, these friends of mine are the people who used to live and to go to the Condorito buildings during the last three decades.

Finally, I would like to thank my special wife Laura Saiter Santos Fardin who was always by my side in the great and worst moments. She has sacrificed her career for a period to accompany me. Also, I thank my mother Ivete Pavesi Fardin, my father Arnóbio josé Fardin, my brother Felipe Pavesi Fardin, his wife Barbara Petroneto Fafa Fardin and my wife's family. Without them, I couldn't overcome all the obstacles. Another special person

Bernardo Bastos de Siqueira, my dentistry office partner since the year 2004 that has encouraged me to face the challenges. How about Alvareta Schneider! This one has a big heart. Thanks for taxing and calling me when you were having a greating time in Espírito Santo State.

Also, I would like to thank the Fundação de Amparo a Pesquisa do Estado de São Paulo (FAPESP) with the specific grant numbers 2011/23177-8; 2013/20806-0 and 2014/22859-6. Only with this financial support I could conclude my researches at the University of São Paulo – Bauru College of Dentistry, Bauru, SP, Brazil and at the Department of Biomaterials and Biomimetics of New York University, NY, USA (1-year BEPE).

ABSTRACT

Residual stress within the porcelain veneer of 3-unit zirconia fixed dental prostheses with different framework designs measured by nanoindentation

The present study aimed to identify different concentrations of residual stress of surfaces of porcelain veneer (PV) fused to zirconia 3-unit fixed dental prostheses (FDPs) with even thickness and modified (lingual collar connected to proximal struts presenting 12 mm^2 connector area) framework designs by nanoindentation method. Twenty-three FDPs replacing second premolar (PM) were fabricated and the cyclic loading was applied on twenty FDPs. Fractured, suspended and non-fatigued FDPs were selected and divided (n=3/each) into: 1) Fractured even thickness (ZrEvenF); 2) Suspended even thickness (ZrEvenS); 3) Fractured with modified framework (ZrModF); 4) Suspended with modified framework (ZrModS); 5) Non-fatigued even thickness (Control). Moreover, the control group surfaces could be divided (n=3/each) into: 6) Mesial PM abutment (MPMa); 7) Distal PM abutment (DPMa); 8) Buccal PM abutment (BPMa); 9) Lingual PM abutment (LPMa); 10) Mesial PM pontic (MPMp); 11) Distal PM pontic (DPMp); 12) Buccal PM pontic (BPMp); 13) Lingual PM pontic (LPMp); 14) Mesial molar abutment (MMa); 15) Distal molar abutment (DMa); 16) Buccal molar abutment (BMa); 17) Lingual molar abutment (LMa). The PV surfaces were nanoindented in regions of interest (ROI) 1, 2 and 3, which were 0.03 mm, 0.35 mm and 1.05 mm from outer PV surface surface towards the PV/framework interface, respectively. Each ROI received 5 nanoindentations with 10 μm of minimum separation loaded to a peak load 4 μN. The Linear Mixed Model test and Least Significant Difference (95%) were used. The statistical analysis among ZrEvenF, ZrEvenS, ZrModF, ZrModS, and Control groups showed differences (p=0.000) except for the comparison between ZrModS and Control group (p=0.371). Also, ROI 1, 2, and 3 were different (p<0.001) with higher residual stresses in outer PV regions relative to those closer to the framework. The comparison among crowns showed that pontic was different from premolar (p=0,001) and molar (p=0,007) abutments, always showing higher residual stress levels. When marginal ridges groups (MPMa, DPMa, MPMp, DPMp, MMa, DMa) were compared, the DMa group was different from DPMp (p=0,004) and MPMa (p=0,00) group, whereas MPMa was different among all groups. The residual stress of porcelain veneer FDPs was different between: fractured and suspended FDPs regardless of the framework design; ROI 1, 2 and 3; and pontic and abutment crowns. Moreover, the proximal areas presented the highest concentration of residual stress.

Key words: Fatigue. Dental porcelain. Dental stress analysis. Denture, partial.

RESUMO

Estresse residual na porcelana de revestimento de próteses parciais fixas de 3 elementos com diferentes desenhos de infra-estrutura mensurado por nanoendentação

O presente estudo teve como objetivo identificar diferentes concentrações de tensão residual de superfícies da cerâmica de revestimento (CR) de próteses parciais fixas de zircônia de 3 elementos (PPFs) com desenho da infraestrutura de espessura mínima e modificada (cinta lingual conectada aos postes proximais com 12 mm^2 de área de conector) pelo método de nanoindentação. Vinte e três PPFs repondo segundo premolar (PM) foram confeccionados e o carregamento dinâmico foi aplicado em vinte PPFs. As PPFs fraturadas, suspensas e não fadigadas foram selecionadas e divididas (n=3/cada) nos seguintes grupos: 1) Fraturadas com espessura mínima (ZrEvenF); 2) Suspensas com espessura mínima (ZrEvenS); 3) Fraturadas com infraestrutura modificada (ZrModF); 4) Suspensas com infraestrutura modificada (ZrModS); 5) Não fadigadas com espessura mínima (Controle). Além disso, as superfícies do grupo controle foram divididas (n=3/cada) nos seguintes grupos: 6) Mesial do pilar PM (MPMa); 7) Distal do pilar PM (DPMa); 8) Vestibular do pilar PM (BPMa); 9) Lingual do pilar PM (LPMa); 10) Mesial do pôntico PM (MPMp); 11) Distal do pôntico PM (DPMp); 12) Vestibular do pôntico PM (BPMp); 13) Lingual do pôntico PM (LPMp); 14) Mesial do pilar molar (MMa); 15) Distal do pilar molar (DMa); 16) Vestibular do pilar molar (BMa); 17) Lingual do pilar molar (LMa). As superfícies da CR foram nanoendentadas nas regiões de interesse (ROI) 1, 2 e 3, a qual a distância da superfície externa da CR no sentido da interface CR/infraestrutura era de 0,03 mm, 0,35 mm e 1,05 mm, respectivamente. Cada ROI recebeu 5 nanoendentações com espaço mínimo de 10 µm para carregar até 4 µN. O teste modelo linear misto com diferença estatística mínima (95%) foi executado usando os valores de dureza. A análise estatística entre os grupos ZrEvenF, ZrEvenS, ZrModF, ZrModS, e Control apresentou diferenças (p=0.000) exceto para a comparação entre os grupos ZrModS e Control (p=0.371). Também, ROI 1, 2 e 3 foram diferentes (p<0.001). A comparação entre coroas mostrou que o pôntico foi diferente dos pilares pré-molar (p=0,001) e molar (p=0,007), sempre apresentando os maiores níveis de tensão. Quando os grupos das cristas marginais (MPMa, DPMa, MPMp, DPMp, MMa, DMa) foram comparadas, o grupo DMa foi diferente dos grupos DPMp (p=0,004) and MPMa (p=0,00), enquanto que o grupo MPMa foi diferente de todos os grupos. A tensão residual da cerâmica de revestimento foi diferente entre: PPFs fraturadas e suspensas independente do desenho da infraestrutura; ROI 1, 2 e 3; e pônticos e pilares. Além disso, as áreas proximais apresentaram a maior concentração de tensão residual.

Palavras-chave: Fadiga. Porcelana dentária. Análise do estresse dentário. Prótese parcial.

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1 Introduction

1 INTRODUCTION

The advances of the esthetic materials have increased their use as an alternative of restorations for missing teeth in anterior and posterior regions. However, the fixed dental prostheses (FDP) of porcelain fused to zirconia (Y-TZP) continue presenting high rates of technical complications (1-5). The porcelain cohesive fracture is reported as the one of the most common complications, reported in a recent systematic review, with cumulative 5 years rate estimated in 34,9% (6). The reasons for such high percentage of failures are still under debate, but have been often associated, among others, to the presence of residual stress generated in the porcelain veneer during the manufacturing process (7-9). In order to decrease its presence, slow cooling rates of the porcelain veneer (8), compatibility in coefficient of thermal expansions between porcelain veneer and zirconia framework (8, 10), framework and connector designs (11, 12) have been recommended. Nevertheless, the understanding of the distribution and magnitude of residual stress in porcelain fused to zirconia prostheses remains limited(9).

Currently, the literature presents various methods to identify and measure residual stress in porcelain veneer, such as: the Vickers indentation (9, 13, 14), finite element analysis (15, 16). Analytical models (8, 17), the hole-drilling method (18, 19), and optical birefringence technique (20, 21). Notwithstanding the methods above, the nanoindentation test has been highlighted in the measurement of residual stress. Its versatility offers potential advantages, such as quantitative indentation to extract local and volume average of hardness, Youg’s modulus, among others, from thin films and other small volumes materials with non-destructive methodology (7, 22). In addition, this method is not limited to simple bilayer geometries, as bars, disks specimens (23), therefore the residual stress of FDP’s important areas can be measured (23). The residual stress can be evaluated by nanoindentation method based on the assumption that an extra force is added to indentation force. As such, if the residual stress is tensile, the extra force acts in the same direction as the indentation force, in other words, the specific nanoindented area would show less resistance to nanoindentation, and consequently the hardness would present lower values. The opposite behavior is expected for compressive residual stress (9).

Previous studies have shown that porcelain veneered zirconia prostheses present higher residual stress in curve areas (20). As such, the proximal areas of crowns (24, 25) and connector gingival areas of fixed dental prostheses (11, 26) have become a critical area. Despite the fact that manufacturer's recommendations have been followed by most dental technicians, these areas often challenging to the longevity of the restoration (27). To avoid porcelain fractures, different framework designs have been suggested for increasing the porcelain veneer support and reduce the extent of porcelain veneer fractures (28, 29). When this approach is applied to crowns, proximal struts connected to lingual collars have increased the fatigue life of crowns in laboratory testing (28, 29), whereas the same improvements are yet to be confirmed for 3-unit porcelain veneered fused to Y-TZP.

Besides the manufacturing process, some findings have important implications on the clinical location of occlusal contact in mouth (27). Surprisingly, the most common occlusal contact in mouth is cusp to marginal ridges (34,60%) (30), where the presence of one to three contacts is commonplace (31). When the occlusal contact involves the marginal ridges of the FDPs, the possibility of porcelain veneered and framework fractures may increase. Once the marginal ridge of the FDP's most proximal areas presents porcelain fracture, the repair with composite resin may be successful or not. Usually, the unsuccessful repair consequences may create a space between both teeth, which allows food accumulation (27). However, the worst scenario would be the FDPs framework fractures often related to the occlusal contact on marginal ridges located at the connector area, which requires their replacement (4, 32).

Accordingly, the purpose of the two studies that constitute this thesis was to identify the presence of different concentrations of residual stress based on hardness, as previously reported by Zhang et al. (9), in different locations of fatigued and non-fatigued 3-unit porcelain-veneered fused to Y-TZP with different framework designs measured by nanoindentation. Four hypotheses were tested: (1) the fractured and suspended fatigued specimens would not present different residual stresses between the same framework design groups, (2) the indented regions located at the outer surface of the porcelain veneer (region of interest – ROI 1) will present higher residual stress compared to the indented regions closer to the framework/porcelain veneer interface (ROI 2 and 3), (3) the connector marginal ridges would present higher residual stress when compared to all surfaces, and (4) the porcelain veneer of the pontic would present higher residual stress compared to the FDPs abutments.

2 Articles

2 ARTICLES

The article 1 presented in this thesis was currently submitted in the *Journal of the Mechanical Behavior of Biomedical Materials* (Annex 1) and the article 2 was written according to the Journal of *Dental Materials*. The first article had the *Declaration of Exclusive Use of the Article in Thesis* signed by all co-authors (Annex 2).

2.1. ARTICLE 1

Residual stress of porcelain-fused to zirconia 3-unit fixed dental prostheses measured by nanoindentation

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ABSTRACT

Objective: To evaluate the residual stress (nanoindentation based on hardness) of fatigued porcelain-fused to zirconia 3-unit fixed dental prostheses (FDP) with different framework designs.

Method: Twenty maxillary 3-unit FDP replacing second-premolar (pontic) were fabricated with conventional framework-design (even-thickness of 0.5 mm and 9 mm^2 connector area) and modified framework-design (thickness of 0.5 mm presenting lingual collar connected to proximal struts and 12 mm^2 connector area). Connector marginal ridges were loaded and the fractured and suspended FDPs were divided (n=3/each) into: 1) Fractured zirconia even-thickness (ZrEvenF); 2) Suspended zirconia even-thickness (ZrEvenS); 3) Fractured zirconia with modified framework (ZrModF); 4) Suspended zirconia with modified framework (ZrModS); 5) Non-fatigued FDP with conventional framework design (Control). The FDPs were nanoindented at 0.03 mm (Region of Interest (ROI) 1), 0.35 mm (ROI 2) and 1.05 mm (ROI 3) distances from porcelain veneer outer surface with peak load 4 μN. The Linear Mixed Model test and Least Significant Difference (95%) were used.

Result: Highest rank hardness values were found for Control group and ZrModS presented the highest hardness values, where the lowest values were found in ZrModF. Statistical differences (p=0.000) were found among all groups except for comparison between ZrModS and Control group (p=0.371). Hardness between ROIs were statistically significant different (p<0.001) where ROI 1 presented the lowest values.

Significance: Framework-design modification did not influence the residual stress of porcelain-fused to zirconia fatigued 3-unit FDP. Whereas fractured FDPs showed the highest residual stress compared to suspended and control FDPs. Residual stress increased as nanoindented away from framework.

Keywords: Fatigue, Porcelain, Zirconia, Nanoindentation, Residual stress, Fixed Dental Prostheses.

1. Introduction

The esthetic benefits and increasing strength of all-ceramic restorations have encouraged their use in the rehabilitation of partially edentulous cases. However, several years after improvements and launch of new systems, all types of all-ceramic fixed dental prostheses (FDP) still present significantly lower survival rates than metal-ceramic systems [1, 2]. Regarding the specific outcomes of porcelain-fused to zirconia (PFZ) prostheses it is remarkable that the variability in results from clinical studies may naturally lead professionals to question the indication safety. The reasons for failure are multifactorial and include low fracture toughness and/or the cooling rates of the porcelain veneer [3], thermal expansion mismatch between porcelain veneer and zirconia framework [3, 4], inappropriate framework designs, higher tensile stresses in the connector area and connector design [5, 6], and the human factor linked to laboratory technicians [7].

The manufacturing process of ceramic, glasses and metal is directly related to the presence of tensile stresses. The processing of veneered all-ceramic systems may culminate in the development of tensile and compression areas which may increase or decrease residual stress concentration, respectively [8]. Once manufactured, the presence of residual stresses may facilitate crack initiation and decrease the longevity of restorations.

One interesting aspect is that, whereas a significant amount of research efforts has been put towards the understanding of multifactorial materials related issues, such as processing and handling, less attention has been devoted to patient functional aspects, such as how teeth relate to each other when in contact. The most extensive fractographic study thus far, from clinically failed alumina and zirconia veneered prostheses, has examined the fractured sites and created a classification system to guide clinicians conduct where non-critical fractures can be polished, reshaped, or repaired and the critical ones demand restoration replacement. Interestingly, the critical fractures occurred at proximal areas and originated from occlusal contacts at the marginal ridges. Consequently, the authors systematically suggested the elimination of contacts on mesial and distal marginal ridges because the veneering ceramic is typically unsupported by the framework in this location [9].

Within this context, although the most prevalent teeth contact occurs between functional cusp tips and marginal ridges (34.60%) [10] it is important to acknowledge that occlusal contacts naturally occur on marginal ridges in the dentition, regardless of Angle's occlusal scheme (class I, II or III) [11]. Simulation studies have shown that marginal ridges and proximal areas present high level of stresses specially in molars [12] and second premolars [13], leading some authors to speculate that tooth contacts at marginal ridges may present a higher risk to restoration failure compared to contacts at the central fossa [9]. In FDP, marginal ridge areas are in part supported by connectors, which have shown to present cohesive failures within the veneering porcelain [14-18] or framework catastrophic failures specially if manufacturer's recommendation of connector dimensions are not strictly followed [15]. Considering this aspect, it is noticeable that most laboratory testing of FDPs have limited loading at the center of the pontic [14], disregarding naturally occurring occlusal contacts at the marginal ridges. The same trend has been noticed in crowns where laboratory studies have concentrated indenter load application typically at the central fossa and only recently, a laboratory study switched the standards loading crowns at the marginal ridges [18]. In single crowns, loading at marginal ridges challenges the restoration integrity since resulting proximal failures are commonly unrepairable [9]. In FDP loading the marginal ridges at connector areas (between abutment teeth and pontic) may lead to competing failure modes from the indenter compression zone and tensile originating at the gingival embrasure of the pontic [16, 19]. This assumption warrants further investigation.

Several methods are available to evaluate the presence of residual stress within the porcelain veneer, such as: Vickers indentation [20, 21], hole drilling [22, 23], analytical models [3, 24], finite element analysis [25, 26], optical birefringence technique [27, 28], and electronic strain analysis [29]. The last two decades showed that the indentation method has been widely used to evaluate the porcelain veneer fused to zirconia. Refinement of this technique has led to the development of nanoindentation that presents the advantage of allowing multiple and non-destructive indentations of the same specimen [30]. In addition, the nanoindentation test can establish the distribution of residual stress of real specimens based on the assumption that materials with lower resistance to the indentation force indicate the presence of tensile stress whereas one with compressive residual stress will show higher resistance" [30]. Studies comparing porcelain veneer hardness between samples with residual stress and control samples without have corroborated this assumption validating the method [31, 32].

The present study sought to evaluate the residual stress based on hardness of fatigued 3-unit porcelain-fused to zirconia (Y-TZP) prostheses with different framework designs classified as fractured or suspended after cyclic loading on marginal ridges. Nanoindentation test was used and two hypotheses were postulated: (1) PFZ FDPs subjected to fatigue testing would not present different residual stresses (evaluated by nanoindentation based on hardness) within the porcelain veneer when fractured or suspended and; (2) Residual stress based on hardness would increase proportionally as the porcelain veneer distance from the framework increased.

2. Materials and Methods

2.1 - Sample preparation

An artificial maxillary first premolar (abutment), a second premolar (pontic) and a first molar (abutment) teeth positioned in a mannequin were transferred to a 25 mm diameter PVC tube and embedded with acrylic resin (Jet, Clássico Artigos Odontológicos, São Paulo, SP, Brazil) until 3 mm below the cervical region. An impression was taken from the 3 teeth with laboratory impression material (Zetalabor, Zermack, Badia Polesine, Rovigo, Italy) to copy the anatomy prior to full crown preparation. The second premolar (pontic) was removed with a diamond bur (4138 KG Sorensen, Cotia, SP, Brazil). After preparation, first premolar and first molar abutment teeth presented a 2 mm occlusal reduction, 1.5 mm axial reduction and a deep chamfer margin.

Replicas of the prepared abutments were fabricated by incremental packing and light-curing (Ultralux, Dabi Atlante, Ribeirão Preto, SP, Brazil) of composite resin (Z100 – 3M Oral Care, St Paul, MN, USA) into polyvinyl siloxane impressions (Express – 3M Oral Care, St Paul, MN, USA) of the abutments. The composite resin replicas were stored in distilled water for 30 days to allow hygroscopic expansion and to eliminate some dimensional alteration after FDPs cementation [33, 34].

A total of twenty PFZ maxillary 3-unit FDPs were fabricated where 10 FDPs comprised frameworks with an abutment even thickness of 0.5 mm and 9 mm^2 connector area (group ZrEven, Figure 1A; 1C; 1E), and another ten FDPs presented frameworks with an abutment thickness of 0.5 mm modified by the presence of a lingual collar height of 2 mm connected to proximal struts of 3.5 mm and 12 mm^2 connector area (group ZrMod, Figure 1B; 1D; 1F). The Y-TZP frameworks were milled from pre-sintered blocks (Ceramill ZI 71, 16 mm) using a CAD-CAM system (Ceramil Amanngirbach, Koblach, Austria). After milling, the frameworks were sintered in the Sintramat furnace at 1,500°C for 8 h. The IPS e.Max Ceram Transpaclear (Ivoclar Vivadent AG, Schaan, Liechtenstein) veneering ceramic was hand layered and the firing schedule followed the manufacturer's instructions (furnace opens at 450°C during glaze firing) (Figure 2). An impression of the anatomy of the unprepared teeth previously made with silicone (Zetalabor, Zermack, Badia Polesine, Rovigo, Italy) was then used as a contour guide during porcelain veneering. An approximate final framework and porcelain thickness of 1.5 mm on the axial walls and occlusal surface was obtained (Figure 3).

The Y-TZP intaglio surface was cleaned with 35% phosphoric acid (Ultra-Etch, Ultradent, South Jordan, USA) for 60s, rinsed with water for 30s and cemented on the prepared composite resin replicas with a self-adhesive dual-cure resin cement (RelyX U200 – 3M Oral Care, St Paul, MN, USA) under a 5 Kg static occlusal load for 10 min. Samples were stored in 37^{0}C distilled water for 7 days prior to testing.

2.2 - Fatigue test

Twenty FDPs were subjected to cyclic loading (Model MSFM – Elquip – São Carlo, SP, Brazil) in water for 2 million cycles or until fracture on each marginal ridge separately (first premolar and second premolar / second premolar and first molar) with a load range from 30-300 N, at 2 Hz with a 3.18 mm radius lithium disilicate indenter at 37°C (Figure 4A; 4B). The test was interrupted every 125,000 cycles for damage inspection at a stereomicroscope (Leica MZE, Mannheim, Germany) coupled to an external light source of two beams (Leica CL5 150D, Mannheim, Germany). Following inspection, specimens and indenters were repositioned to continue fatigue testing.

2.3 – Failure mode characterization

Fractures of the porcelain veneer and/or framework were used as failure criteria. If marginal ridges of FDPs survived 2 million cycles not presenting such fractures (porcelain veneer contour anatomically preserved) they were classified as suspended FDPs. The Scanning Electron Microscopy (SEM) (Model S-3500N; Hitachi, Japan) was used for fractographic analysis of the most representative chipping fractures. In addition, suspended FDPs not used for nanoindentation, were embedded in epoxy resin (Resina Epoxi RD6921, Redelease, São Paulo, Brazil), precision sectioned with diamond saw and then polished with silicon carbide papers (320, 400, 500, 600, 1200, 2000 and 2500 grits), under copious water irrigation for near and far-field damage inspection under stereomicroscopy.

2.4 – Nanoindentation test

Fatigued FDPs were inspected under stereomicroscopy and subdivided (n=3 each group) for nanoindentation testing according to the presence or absence of fracture, as follows: 1) Minor cohesive fracture within the porcelain veneer (≤ 4 mm^2) in FDPs with even thickness frameworks (ZrEvenF); 2) Absence of fracture (suspended) in FDPs with zirconia even thickness frameworks (ZrEvenF); 3) Minor cohesive fracture within the porcelain veneer in FDPs with modified framework (ZrModF); 4) Suspended FDPs with modified framework (ZrModS), and; 5) Non-fatigued FDP with conventional framework design (Control).

A 0.7 mm diameter metallic wire was positioned on the central sulcus of the FDP crowns prior to its embedding in epoxy resin. After setting, the metallic wire served as reference for positioning the FDP before sectioning with a precision diamond saw (Isomet 2000, Buehler, Lake Bluff, IL, USA) in the axial plane. Sectioning was performed under copious water irrigation, parallel to the occlusal plane guided by the metallic wire, which generated a flat section for nanoindentation testing.

The sectioned piece was bonded to an acrylic plate with acrylate-based cement (Fisher Scientific, Waltham, MA, USA). After a setting time of 24 h, the slide containing the sectioned FDP was grounded (400 to 1200 grits Silicon carbide abrasive paper) until 1 mm below the metallic wire, under copious water irrigation, followed by polishing (diamond suspensions of 9 to 1 µm particle size) (Buehler, Lake Bluff, IL, USA). The surface was inspected by means of a stereomicroscope in order to avoid the nanoindentation test on flawed surface areas.

Nanoindentation testing was performed at room temperature in ambient air (± 23 ºC). A nanoindenter (TI 950 TriboIndenter, Hysitron, Minneapolis, MN, USA) equipped with Berkovich diamond three-sided pyramid was used to indent the porcelain veneer under dry condition in the buccal, lingual, mesial, and distal regions of the pontic crown and the abutments (Figure 6). The outer surface of the porcelain veneer determined the distances between the three indentation ROIs: ROI 1, ROI 2, and ROI 3 indentations were performed at 0.03 mm, 0.35 mm, and 1.05 mm from the outer surface of the porcelain veneer towards the Y-TZP framework, respectively (Figure 6). Each ROI received five nanoindentations with 10 µm of minimum separation (Figure 6). The sample was loaded to a peak load 4 µN and unloaded. The hardness was given by:

$$H = \frac{Pmax}{A} \quad \text{(Equation 1)}$$

where H is the hardness value in pascal (Pa) measurement unit, Pmax is the maximum applied force in micronewton (µN) measurement unit and A is the project contact in nanomilimiter (nm) measurement unit. The values are directly obtained from nanoindentation software.

2.5 – Data analysis

The Linear Mixed Model test and Least Significant Difference for multiple comparisons) with mean rank and confidence intervals (95%) were performed using the Statistical Package for the Social Sciences (SPSS) v.23.0 (IBM Corp., USA) software to compare residual stresses based on hardness of fractured and suspended fatigued 3-unit FDPs. Moreover, residual stresses at different nanoindentation ROIs (0.03 mm, 0.35 mm and 1.05 mm) were also compared. Then, plots were created by Origin Pro 2015 software.

3. Results

The plot shown in figure 7-A presents the total sum of rank hardness values for each group. Note that low hardness values represent high concentration of residual stress, since hardness values are inversely proportional to residual stress levels. Residual stress levels were statistically different (p=0.000) among groups except for the pairwise comparison between the Control group and ZrModS group (p=0.37) which presented the highest hardness values (Figure 7 A). The lowest hardness values were observed for ZrModF, followed by ZrEvenF, and ZrEvenS. In general, control and suspended samples presented significantly higher hardness values compared to fractured samples. Detailed nanoindentation values per group are presented in Table 1 A.

The rank hardness values of different nanoindentation ROIs at the porcelain veneer showed statistically significant differences (p<0.001) between all ROIs and groups. A general trend in the presence of highest residual stress were observed for nanoindentation ROI 1 (porcelain veneer away from the framework) irrespective of group (Figure 7 B). Residual stresses significantly decreased as nanoindentations ROIs approached the porcelain veneer/framework interface (detailed values in Table 1 B).

3.1 – Failure modes

Minor porcelain cohesive fracture (≤ 4 mm^2) was the most common failure mode during fatigue. Out of 10 fatigued FDPs of ZrEven group, three presented fractures occurring at 125,000, 625,000, and 1,000,000 cycles and the other 7 were suspended. Out of 10 fatigued FDPs of ZrMod, 4 presented fractures at 125,000, 175,000, 1,250,000, and 2 million cycles, and the remaining 6 were suspended. Fractures between groups were not different in size and type. No Y-TZP framework fractures were observed.

Quasiplastic deformation after the first 125,000 cycles at the indentation contact occlusal surface was observed with both Y-TZP groups. Fractographic marks including hackles, wake hackles, twist hackles, and arrest lines suggested that the crack initiated at the indentation area and propagated towards the margins of the fractured surface in all groups (Figures 8 and 9).

Suspended FDPs that were embedded for damage inspection (not used for nanoindentation) showed the consistent development of inner cone cracks due to cyclic fatigue at the occlusal surface, regardless of framework design (previous Figure 4C, 4D, 4E, and 4F). Interestingly, besides the near-field inner cone cracking, the presence of a competing failure mode depicted by crack development at the gingival portion of the connector area of a ZrEven FDP was documented (Figure 10).

4. Discussion

Considering that porcelain-fused to zirconia FDPs either fractured or suspended after fatigue did present significant differences in hardness within the porcelain veneer our first hypothesis is rejected. The highest hardness values and allegedly lowest residual stresses were observed in the control non-fatigued and in the fatigued non-fractured FDPs with a modified framework design. In contrast, significantly higher residual stress based on hardness was observed for all other fatigued FDPs, increasing significantly for suspended and then for fractured even thickness FDP with the highest levels for fractured FDPs with a modified framework design. Our paired comparison showed contrasting results where fatigued FDPs with a modified framework design presented less residual stress only when suspended, but higher than even thickness FDPs fractured during fatigue. The residual stress results from the detailed analysis within the porcelain veneer may help explain these findings where residual stresses were significantly lower along the depth of all indentation ROIs of the modified compared to the even thickness frameworks, suggesting that the increased bulk of porcelain veneer thickness resulting from an even thickness framework design was less efficient in accommodating residual stresses even during slow cooling. Based on such findings, our second postulated hypothesis that residual stress based on hardness would increase proportionally as the porcelain veneer distance from the framework increased was accepted and is in agreement with a recent study [35].

Analysis of fractured samples showed high residual stresses for both groups when compared to their suspended counterparts and the control. The ZrEvenF presented 3 times more residual stress compared with the Control group and 2.3 times more compared with ZrEvenS group. Highest levels of residual stresses were observed in FDPs with a modified framework design, which presented 3.5 times more residual stress concentration for ZrModF group compared with ZrModS group. Whereas increased residual stresses values were observed in both fractured compared to suspended FDPs, regardless of framework design, the highest discrepancy between the groups were observed in the presence of framework design modification. This finding may have occurred not only due to the increased bulk of Y-TZP framework for this group eventually being detrimental during the glaze firing considering that it may have exacerbated the low thermal conductivity of Y-TZP (2 $Wm^{-1} K^{-1}$) [3], but also because of the lower thickness of the porcelain veneer, which at the outer surface presented the highest residual stresses. Our fractured samples showed the consistent presence of twist hackles, regardless of framework design, which is a telltale fractographic mark associated with the presence of residual stresses [36].

Fractographic analysis allowed the identification of marks that showed where fracture origin was located and the direction of crack propagation. One interesting finding was the overall fracture sizes between groups that were very similar, which was not expected since an effect of underlying framework support would, as reported previously [37-39], theoretically influence final porcelain cohesive fracture dimension. Although in contrast with previous results, the present findings should be compared with caution since most of cited studies were performed in crowns, where herein and in any FDPs study support from the underlying framework will always be present when loading is performed on marginal ridges instead of on cusps. Another reason that may explain the similar porcelain cohesive fracture sizes, regardless of framework design, was that since a slow cooling protocol was used, most residual stresses likely of a tensile type, were observed in the porcelain outer surface, where most fractures were confined. From a clinical perspective, the reduced fracture sizes may translate to feasible chairside repair, and extended function.

The present findings are in agreement with a study using the hole-drilling technique which measured the stress profile in bilayered discs presenting different thickness of framework and porcelain veneer. Its results showed that compressive stresses were found in the veneering ceramic surface of thicker frameworks and tensile stress were observed in the interior part of thinner framework samples [40]. However, when different thicknesses of veneering ceramic were investigated, the compressive stress located at the ceramic surface for samples veneered with 1.0 mm decreased from the surface at 0.5 mm until 0.7 mm and became more compressive again near to the framework [40]. Although macro indentation [21, 35] and less frequently nanoindentation studies [30] have been used to report residual stresses on veneering porcelains, future studies including in silico and more than one laboratory method are warranted to assess their accuracy. The possibility to perform several nanoindents in this study, whereas time consuming, allowed the evaluation of the variability of residual stresses through several measurements along the veneer/framework interface.

Although great fanfare continues on the indication of Y-TZP for fixed dental prostheses, in spite of evidence from systematic review reporting its inferior survival rates compared to metal ceramics up to 5 years [1, 2], it is apparent that several lessons have been learned and improvements in handling of the material will reveal consistently high survival rates among well designed randomized controlled trials in the near future.

5. Conclusion

Residual stress of porcelain veneer of fractured or suspended fatigued Y-TZP 3-unit fixed dental prostheses varied irrespective of framework design modification even when the slow cooling protocol recommended by the manufacturer was used. The outer layer of the porcelain veneer presented the highest concentration of residual stress which decreased towards the framework/veneer interface.

ACKNOWLEDGEMENTS

This study was supported by Fundação de Amparo à Pesquisa do Estado de São Paulo (São Paulo Research Foundation - FAPESP) grants: 2011/16377-0, 2011/23177-8, 2013/20806-0, 2014/22859-6, 2012/19078-7 - Young Investigators Awards, and **EMU 2016/18818-8.** Also in part by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) grant # 309475/2014-7 and 309475/2014-7.No manufacturer supported this study.

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CAPTIONS TO FIGURES

A	**Groups (Rank.hardness)**									
	Control	upper	**ZrEvenF**	upper	**ZrEvenS**	upper	**ZrModF**	upper	**ZrModS**	upper
		lower		lower		lower		lower		lower
Sum of surfaces and ROI (MPa)	1996,04	2031,76	669,05	704,44	1535,20	1570,60	564,23	599,62	1973,10	2008,48
		1960,32		633,66		1499,81		528,84		1937,71
B	**ROI (Rank.hardness)**									
	Control	upper	**ZrEvenF**	upper	**ZrEvenS**	upper	**ZrModF**	upper	**ZrModS**	upper
		lower		lower		lower		lower		lower
ROI 1 (MPa)	1628,09	1690,12	415,41	476,70	1388,35	1449,64	305,60	366,90	1839,17	1900,47
		1566,05		354,12		1327,06		244,30		1777,88
ROI 2 (MPa)	2122,95	2184,99	751,86	813,16	1563,67	1624,96	647,72	709,02	1997,88	2059,17
		2060,92		690,57		1502,37		586,43		1936,58
ROI 3 (MPa)	2237,10	2298,56	839,89	901,17	1653,60	1714,89	739,38	800,67	2082,24	2143,53
		2175,62		778,59		1592,30		678,08		2020,95

Table 1) Pairwise rank.hardness comparison calculation amongst groups (A) and positions (ROI) (B) nanoindented. Note that differences are presented in non-overlap between upper and lower limits.

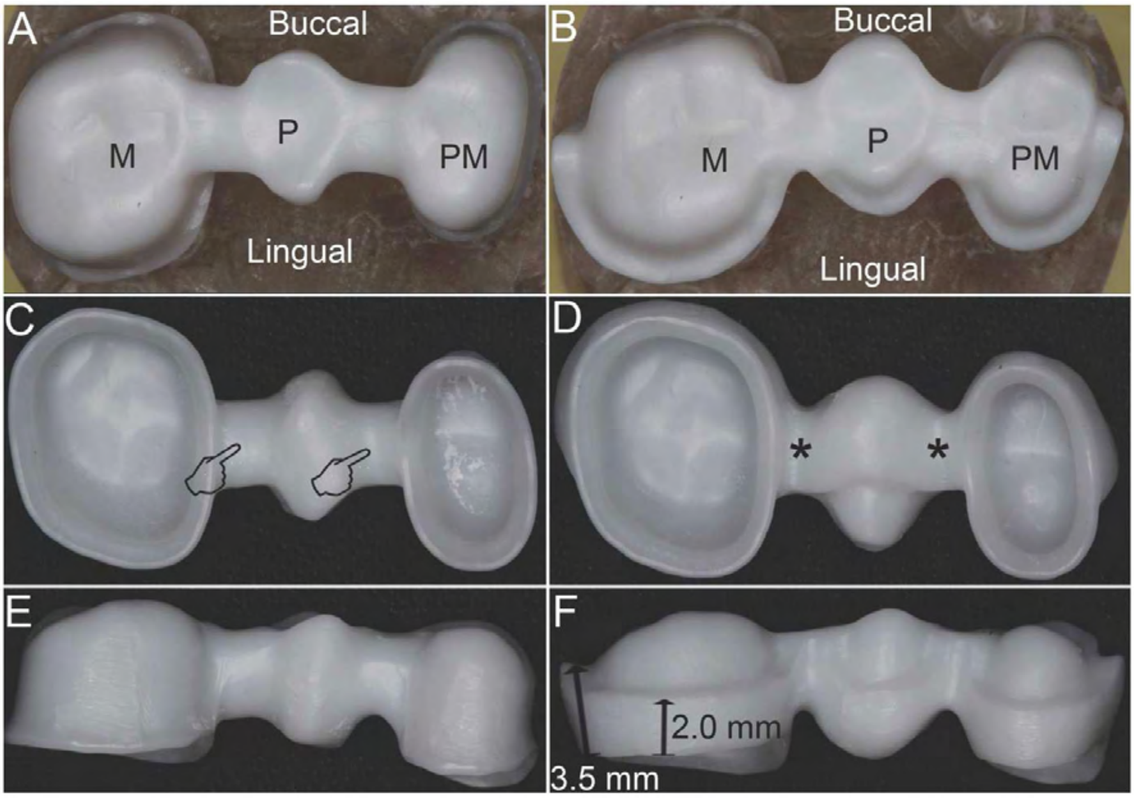

Figure 1) Framework occlusal surface view of 3-unit Y-TZP FDP presenting the first molar (M) and first premolar (PM) as abutments, and second premolar as pontic (P) with different designs: even thickness (A) and modified framework designs (B). The connector gingival surfaces of the Y-TZP even group (pointers) could receive the porcelain veneer layer (C), whereas the Y-TZP mod specimens could avoid the connector gingival stress by the extra extension of the framework at the gingival area (asterisks) (D). Approximate lingual view of the 3-unit Y-TZP frameworks with even thickness (E) and modified with proximal struts and lingual collar (F) and their respective dimensions.

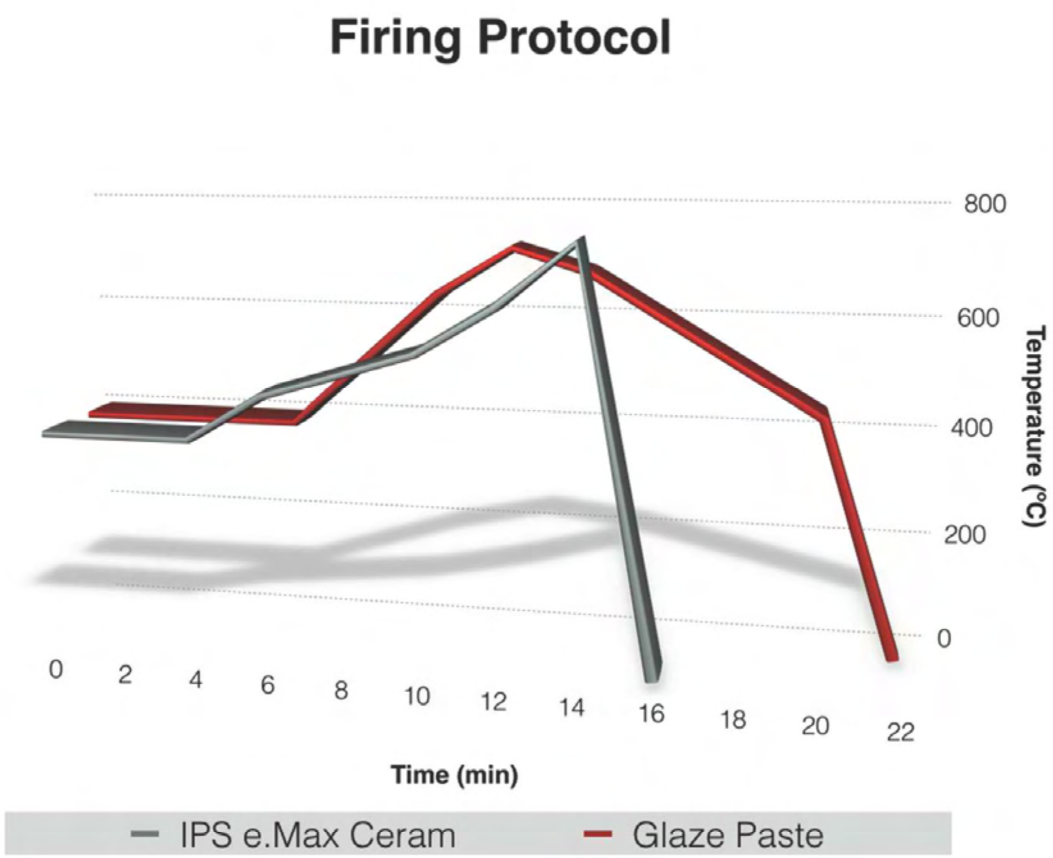

Figure 2) Programat® EP 3000—firing protocols for e.Max Ceram and Glaze paste. The glaze paste firing followed the slow cooling protocol recommended by the manufacturer.

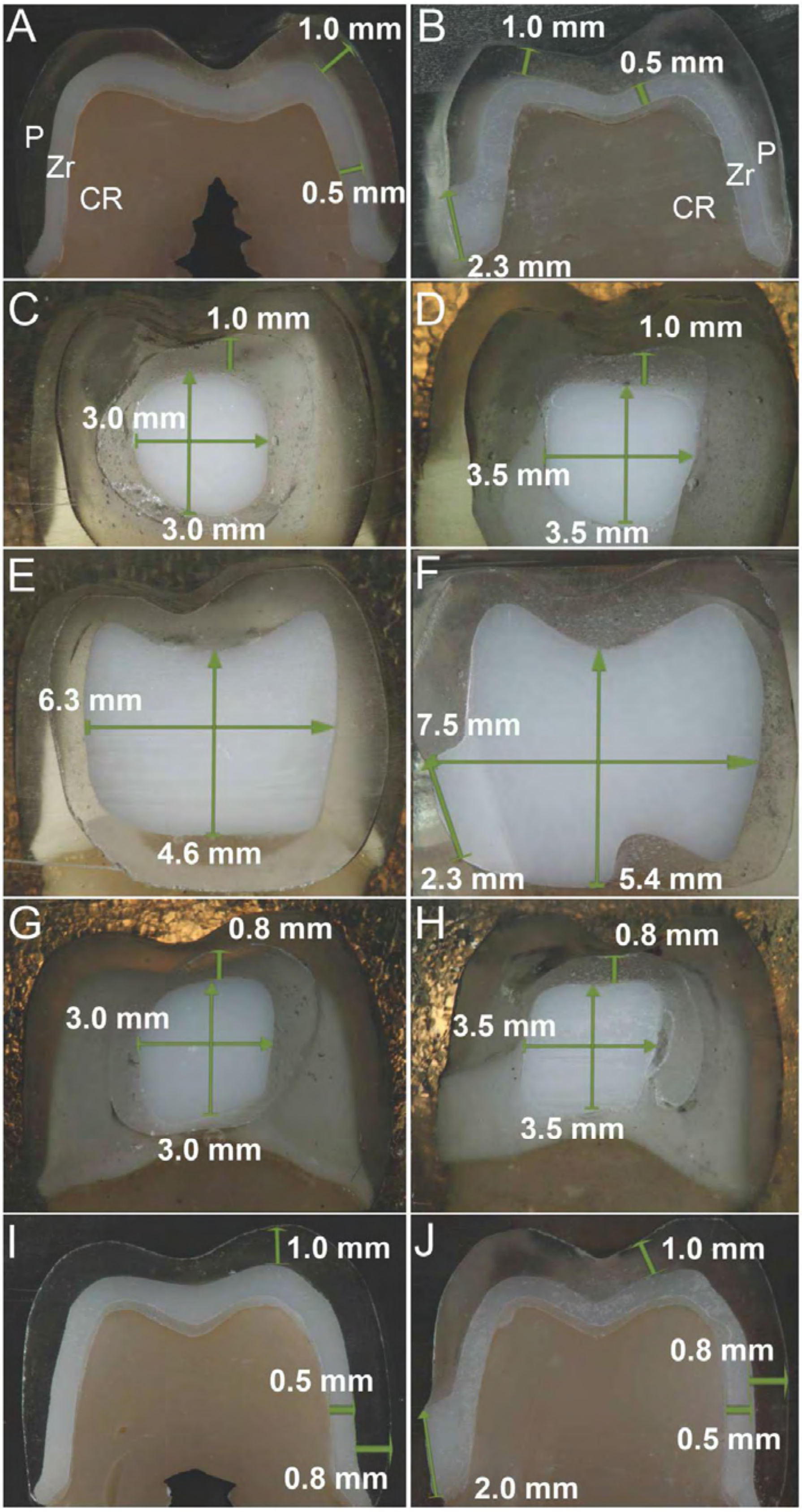
A
1.0 mm
P
Zr
CR
0.5 mm
B
1.0 mm
0.5 mm
P
Zr
CR
2.3 mm
C
1.0 mm
3.0 mm
3.0 mm
D
1.0 mm
3.5 mm
3.5 mm
E
6.3 mm
4.6 mm
F
7.5 mm
2.3 mm
5.4 mm
G
0.8 mm
3.0 mm
3.0 mm
H
0.8 mm
3.5 mm
3.5 mm
I
1.0 mm
0.5 mm
0.8 mm
J
1.0 mm
0.8 mm
0.5 mm
2.0 mm

Figure 3) Dimensions of the frameworks and veneering porcelain in a buccal-lingual section of Y-TZP even (A, C, E, G, I) and Y-TZP mod (B, D, F, H, J). Molar abutment dimensions for Porcelain (P), Zirconia (Zr), cemented onto composite resin core (CR) (A, B). Dimension of the connector located between the distal surface of the first molar (abutment) and the mesial surface of the first premolar (pontic) (C, D). Pontic dimension of the central area of the first premolar (E, F). Dimension of the connector located between the distal surface of the first premolar (pontic) and the mesial surface of the second premolar (abutment) (G, H). Premolar abutment dimensions (I, J)

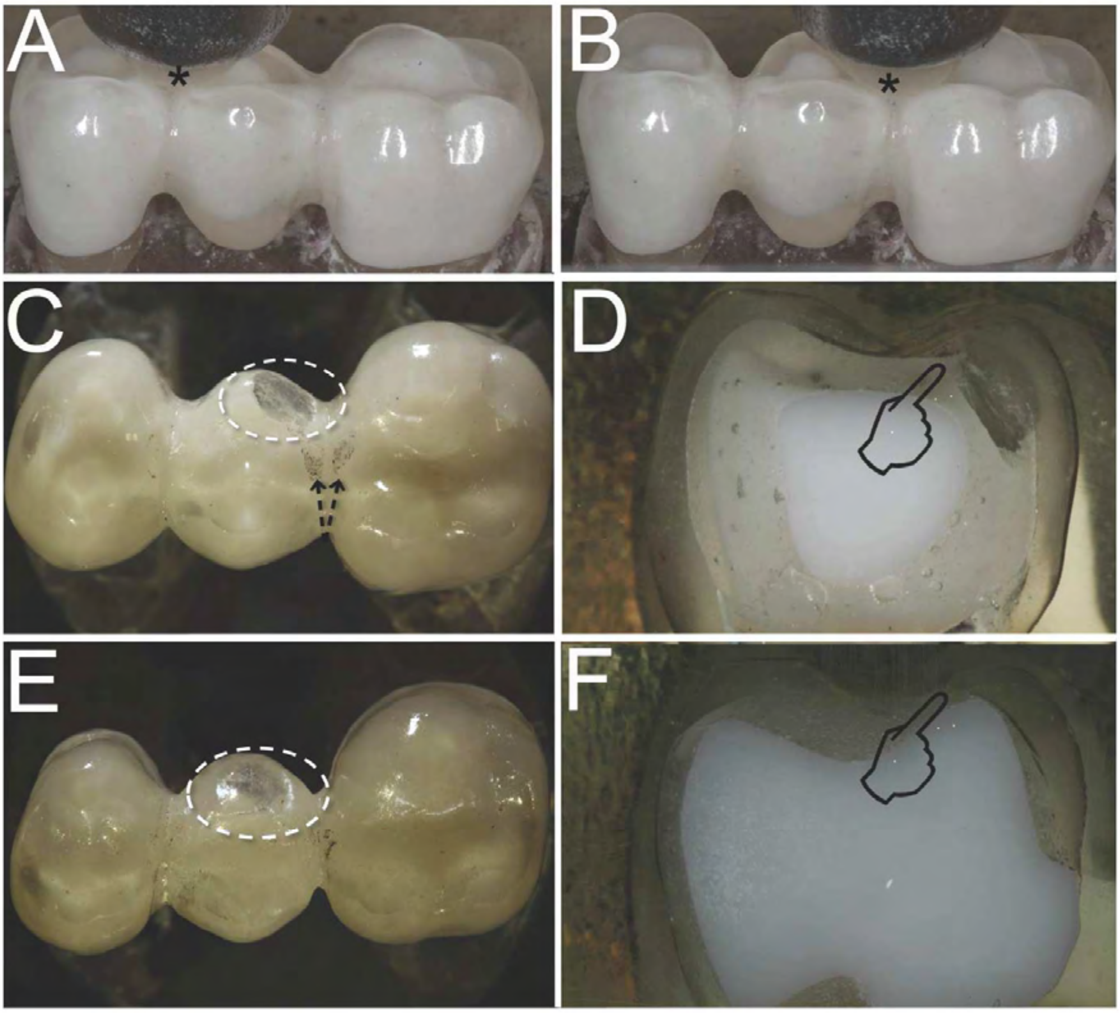

Figure 4) Ceramic indenters (asterisk) positioned in contact with premolar marginal ridges delivery the load until 2 million cycles or fracture (A). Subsequently, the marginal ridges of the second premolar (pontic) and molar (abutment) were also loaded until 2 million cycles or fracture (B). The

suspended specimens presented the contact area marking created by articulating paper (arrows). The cracks present in Y-TZP even (C) and Y-TZP mod (E) were similar. The crack propagation was similar since the origin (pointer) until the ending crack (D) (F).

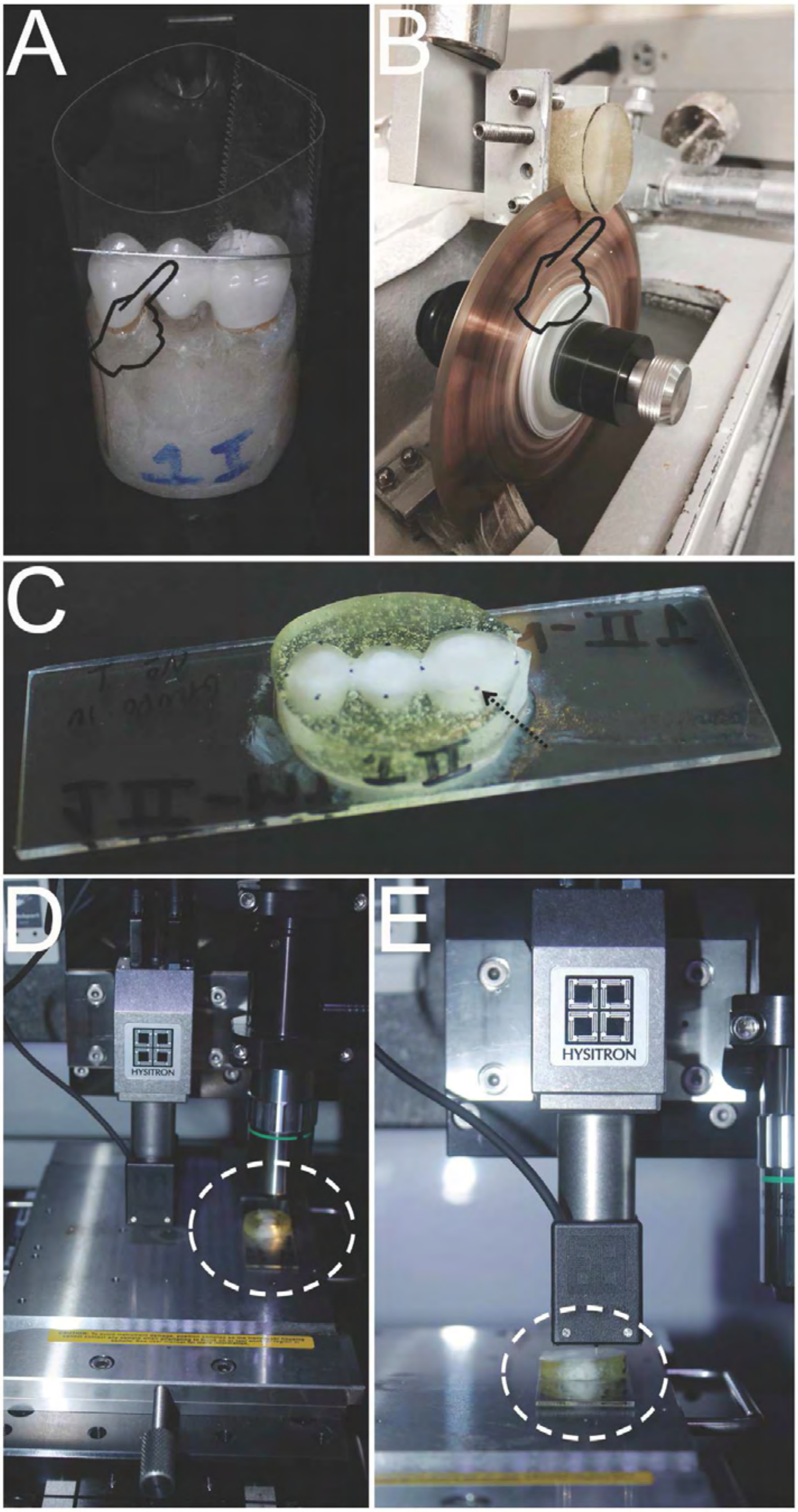
A
B
C
D
HYSITRON
E
HYSITRON

Figure 5) (A) FDP embed in epoxy resin with the metal wire positioned in the center of the sulcus for orientation of the section in the mesio-distal direction (pointer). (B) First section parallel to the occlusal surface (pointer) and the second section in the FDP by means of the diamond saw fixed in Isomet 2000 machine. (C) Approximate view of the specimen showing black marks (arrow) in different surfaces to facilitate the identification of the positions before nanoindentation. (D) Scanning and (E) nanoindenting the sample (circle) using the Hysitron machine.

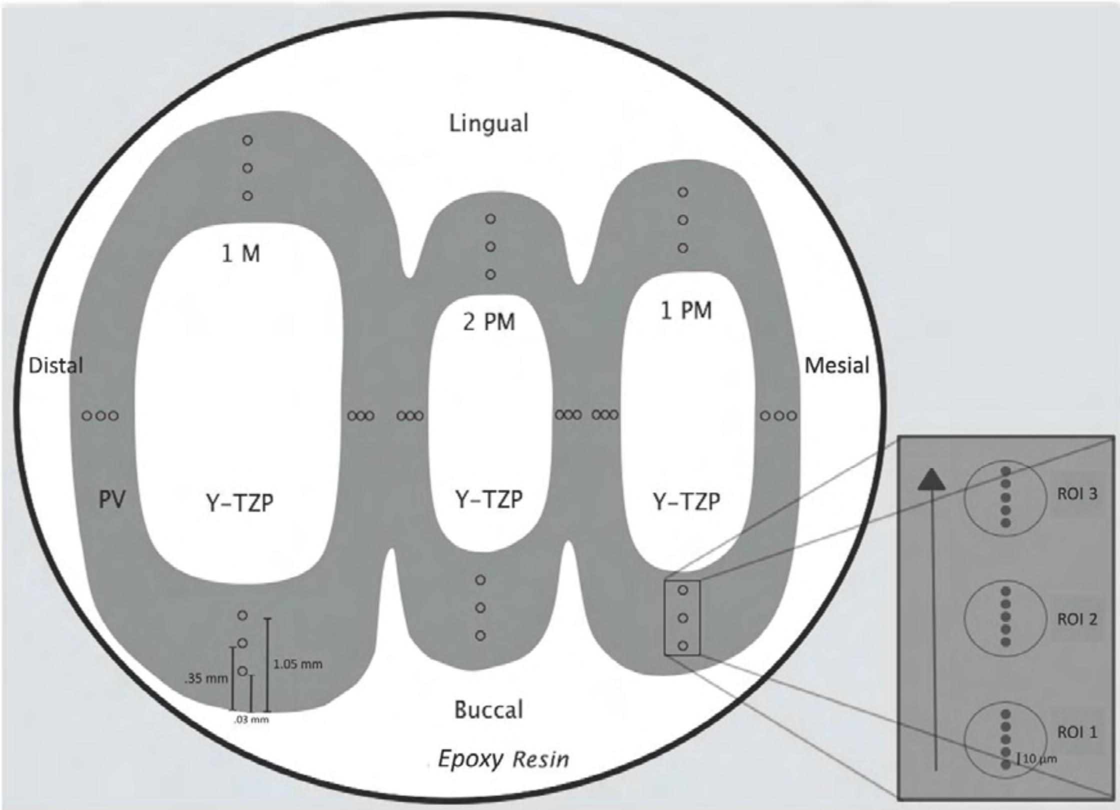

Figure 6) Schematic view showing the areas of analysis of the sectioned sample parallel to the occlusal plane for nanoindentation test. The distances nanoindented was 0.03 mm, 0.35 mm and 1.05 mm from outer porcelain surface for ROIs 1, 2, and 3, respectively. The magnified view showed five nanoindentation points with 10 µm of minimum separation in each ROI. The arrow demonstrated the sequence of the nanoindentation test, which the ROI 3 was always closer to the framework/porcelain veneer interface regardless of the surface. Y-TZP: Zirconia; PV: Porcelain veneer; 1M: First molar; 2 PM: Second premolar; 1 PM: First premolar.

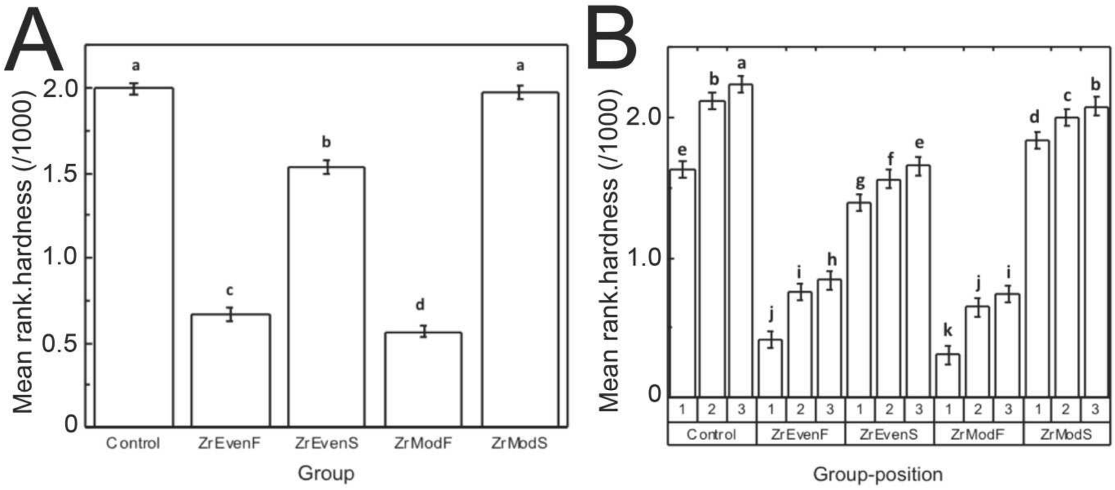

Figure 7) The plot presented letters to clarify where there was statistically difference amongst the groups. (A) Mean rank hardness values (divided by 1000) along five groups presenting the sum total of hardness from different areas (ROIs and surfaces) of abutment and pontic crowns. (B) Mean rank hardness values for the ROIs (1, 2, and 3) regardless of abutment and pontic crown surfaces.

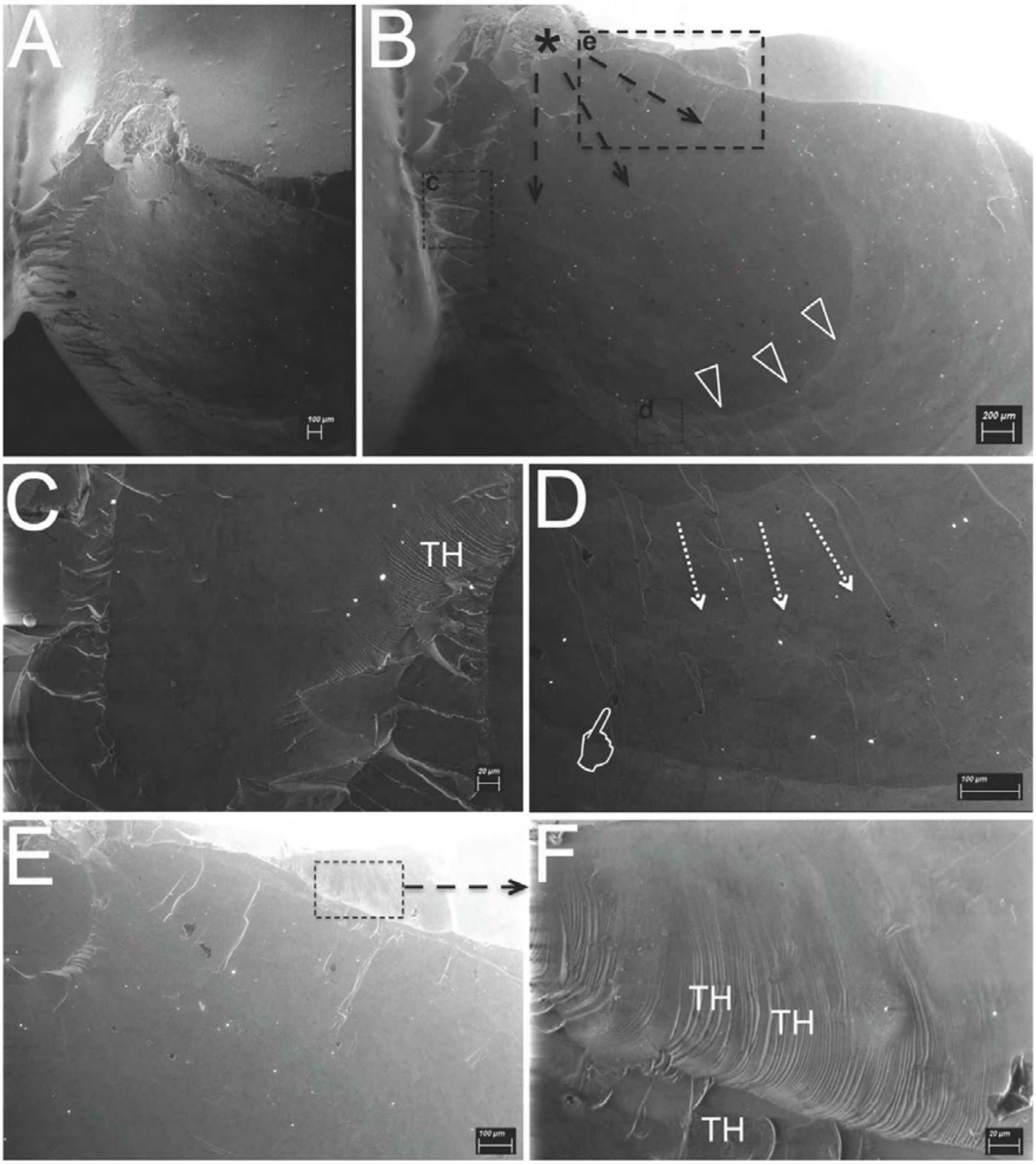

Figure 8) Representative scanning electron micrographs of a specimen from the ZrEvenF group. (A) An occlusal and (B) proximal views of a major porcelain fracture located on molar abutment after 1 million cycles depicting the indentation area (asterisk) and the presence of arrest lines (Δ). The arrows show the direction of the propagation and the squares represents the location of the approximated views in sequence. (C) a magnified view depicts marks such as twist hackles (TH). (D) The origin of the fracture was correlated to the indentation area because several hackles are pointing

towards the cervical margins (arrows). (E) and (F) Twist hackles (TH) which indicate the possible of presence of residual stress and crack direction.

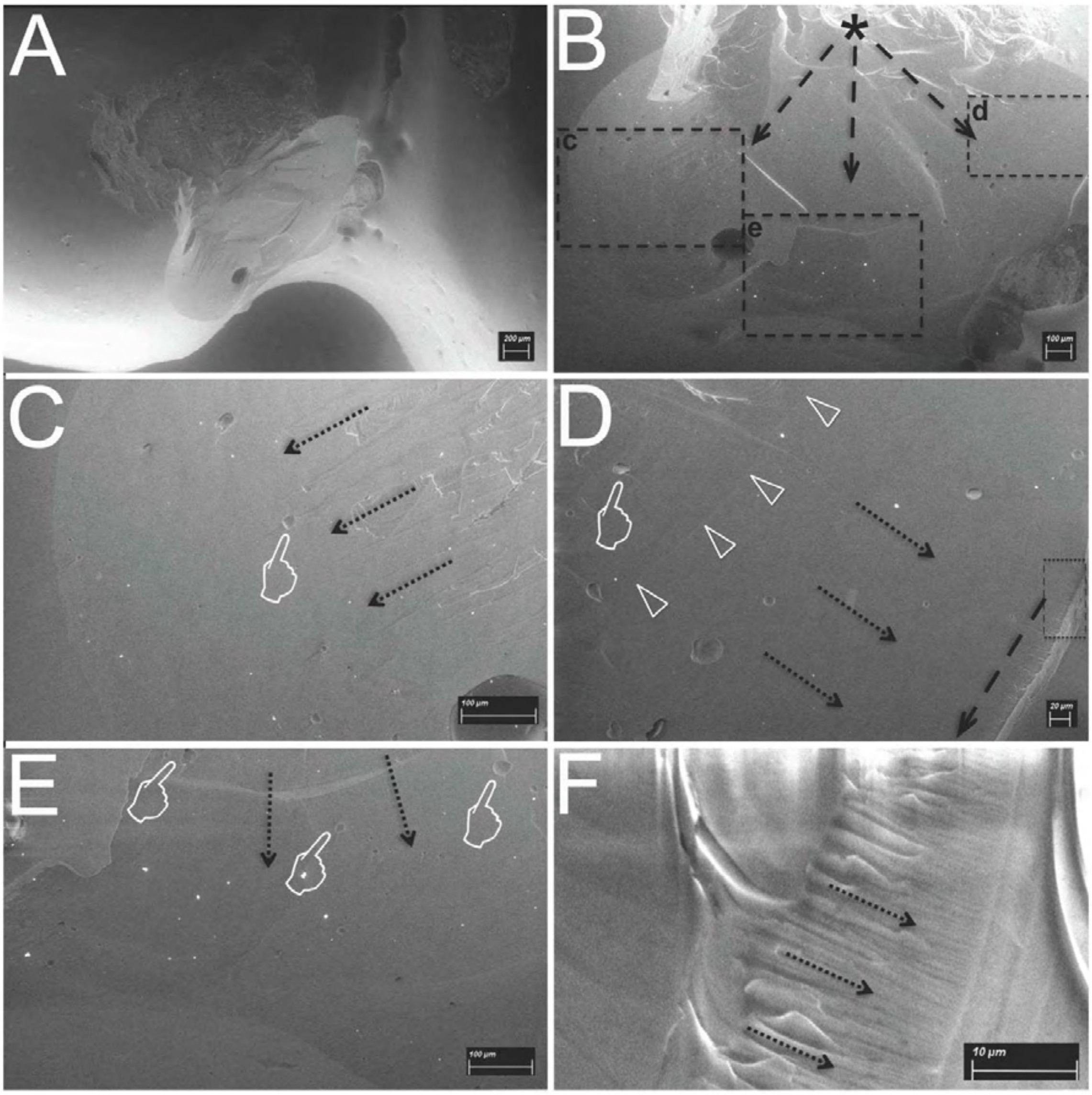

Figure 9) Representative scanning electron micrographs of a specimen from the ZrModF group. (A) An occlusal and (B) proximal views of a minor porcelain fracture located on premolar (pontic) after 125,000 cycles. Also, the indentation area presents quasiplastic deformation (asterisk) and the direction of the fracture propagation is shown by arrows and the squares represent the location of (C), (D) and (E) magnified views. The wake hackles (white pointers) (C; D; E), arrest lines (Δ) (D) and

hackles (C; D; E; F) pointed to proximal areas despited the origin of the fracture was located at the indentation area.

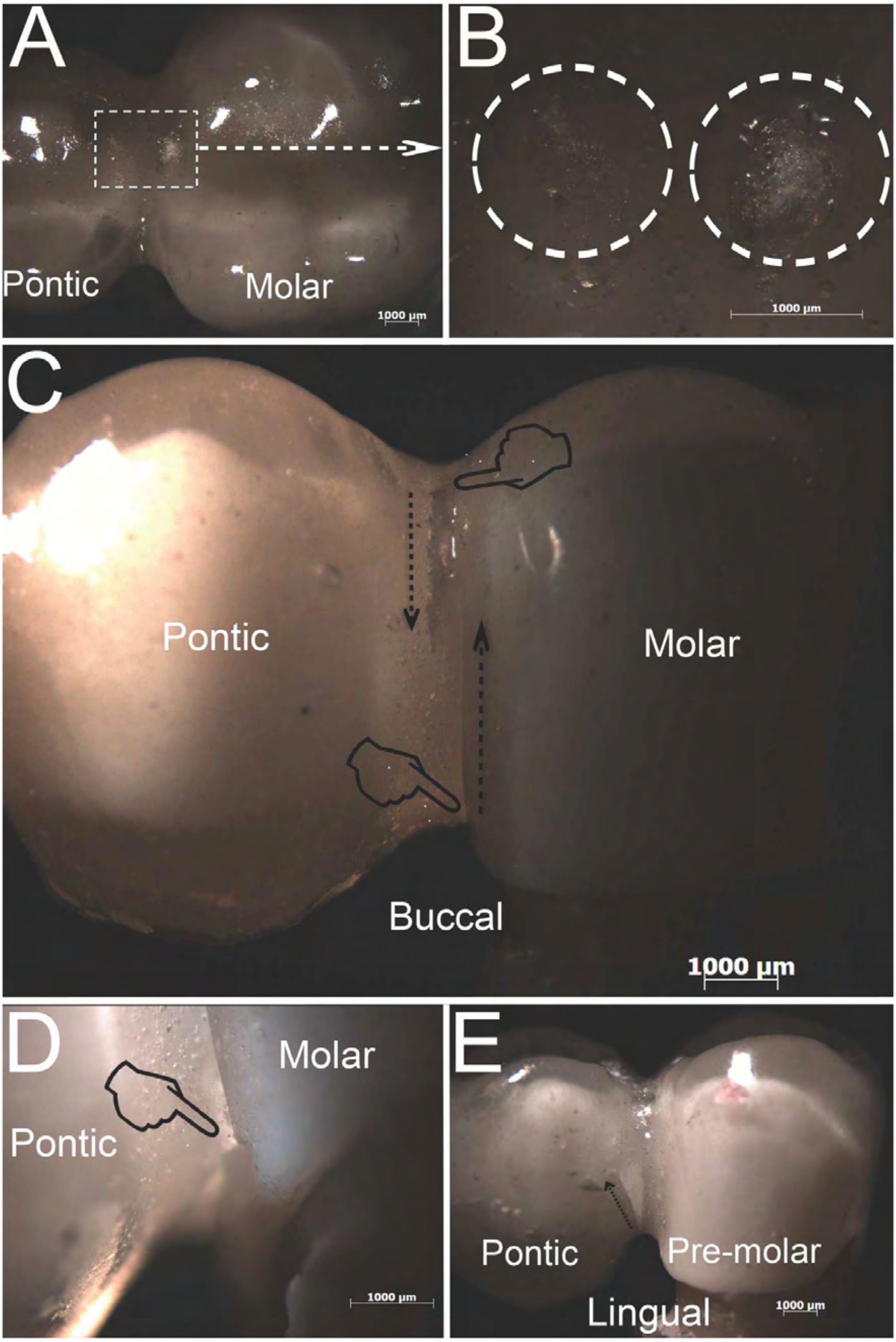
A
Pontic
Molar
1000 µm
B
1000 µm
C
Pontic
Molar
Buccal
1000 µm
D
Molar
Pontic
1000 µm
E
Pontic
Pre-molar
Lingual
1000 µm

Figure 10) Micrographs of suspended ZrEven specimen has shown the indenter contact on the marginal ridges of the pontic and abutment (A). The approximate view of the occlusal surface has shown the presence of quasiplastic deformation (white circles) when loaded by ceramic indenters (B). Competing failure modes were present (arrows) and characterized by different origins of the crack, one coming from the gingival area and the other from occlusal area (pointer) (C). The approximated view of the gingival connector area showed the origin of the gingival crack (pointer) (D), whereas the lingual surface showed the crack origin related to the connector gingival area (E).

2.2. ARTICLE 2

Hardness values on abutments and pontics of porcelain veneer fused to yttria-stabilized zirconium-dioxide (Y-TZP) 3-unit fixed dental prostheses.

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ABSTRACT

Objectives: To identify different concentrations of residual stress based on hardness of porcelain fused to zirconia 3-unit fixed dental prostheses (FDP) in different surfaces of pontic and abutments measured by nanoindentation.

Methods: Three composite-resin replicas of maxillary first premolar and first molar abutment prepared crowns were fabricated to obtain three 3-unit FDPs presenting 0,5 mm thickness and 9 mm^2 connector framework area. The FDP was hand-layered and fired (slow cooling protocol). The FDPs were resin cemented, embedded in epoxy resin, sectioned and polished. The specimens were subdivided (n=3/each) into: 1)Mesial premolar abutment (MPMa); 2)Distal premolar abutment (DPMa); 3)Buccal premolar abutment (BPMa); 4)Lingual premolar abutment (LPMa); 5)Mesial premolar pontic (MPMp); 6)Distal premolar pontic (DPMp); 7)Buccal premolar pontic (BPMp); 8)Lingual premolar pontic (LPMp); 9)Mesial molar abutment (MMa); 10)Distal molar abutment (DMa); 11)Buccal molar abutment (BMa); and 12)Lingual molar abutment (LMa). The porcelain veneer surfaces were nanoindented with peak load 4 µN. The Linear Mixed Model test and Least Significant Difference (95%) were used.

Results: Pontic crowns presented the highest hardness value and was statistical different compared to premolar (p=0.001) and molar (p=0.007) abutments. The comparison among connector and proximal marginal ridges showed that MPMa and DMa groups presented lower hardness values. Moreover, the DMa group was statistically different when compared to DPMp (p=0.004) and MPMa (p=0.000) groups, whereas the MPMa group was statistically different among all marginal ridges groups.

Significance: The 3-unit FDPs residual stress was different between pontic and abutments. The proximal areas presented the highest concentration of residual stress.

Keywords: Fatigue; Porcelain; Zirconia; Nanoindentation; Residual stress; Fixed Dental Prostheses.

1. Introduction

Aesthetic demand has increased the use of all-ceramic systems as an alternative for metal-ceramic ones [1-5]. Additionally, the material's properties improvements have allowed a wide range indication of porcelain fused to zirconia (PFZ) for both anterior and posterior missing teeth due to its high strength and fracture toughness [6]. However, porcelain veneer chippings and large fractures are still reported for Y-TZP system [7], specially at marginal ridges of single crowns [8], and fixed dental prostheses [9]. One commonly associated cause is the presence of residual thermal stress compromising the mechanical resistance of the marginal and proximal areas of molars [10], premolars [11, 12] and connector areas [13]. Additionally, occlusal loading results in damage accumulation, especially when the occlusal contact is located at marginal ridge when an increase in risk of porcelain veneer fracture has been reported [8].

Therefore, studies assessing specific areas of crowns, such as marginal ridges [14] and pontics compared to abutments of FDPs [15] may provide information to whether some sites are more prone to failure. A clinical follow up study of porcelain fused to zirconia FDPs showed that pontics were less involved in dynamic occlusion after cementation [16]. Another clinical study also reported that pontic areas of Y-TZP FDPs presented higher rates of porcelain veneer fractures [9]. This follow up study evaluated 4- to 6-unit porcelain veneer fused to zirconia FDPs (tooth and implant-retained restorations) and described that the chippings location on abutment and pontic crowns was 20% and 80% for tooth retained restorations, respectively. The implant-retained prostheses presented the chipping prevalence of 55% for abutment crown chipping and 45% for pontic crown.

Previous studies have proposed that residual stress is the key factor in avoiding porcelain fractures. Besides the occlusal contacts, the literature shows some factors associated with porcelain veneer fractures including: low fracture toughness, inappropriate framework support, thermal expansion mismatch between porcelain veneer, zirconia framework, and veneer porcelain cooling process [6, 17]. Even when all variables are well controlled, most manufacturers have recommended a slow cooling protocol for porcelain veneer fused to zirconia at the glazing firing stage. The rationale behind it includes the low thermal diffusivity of zirconia, which generates an elevated temperature gradient, specifically in fast cooling protocols [18]. However, the slow cooling protocol does not seem to guarantee an even distribution of residual stress at all surfaces of the porcelain veneered onto zirconia frameworks. This observation has been explained by the presence of curved porcelain-zirconia interface, which supports a different residual stress distribution [19, 20]. Therefore, residual stress distribution in anatomically-relevant specimens, such as 3-unit FDPs, is yet to be investigated.

A variety of methods for measuring residual stresses have been used for bilayered materials. The conventional methods are as follows: Vickers indentation [15, 21], hole drilling [22, 23], analytical models [6, 24], electronic strain analysis [25], finite element analysis [20, 26] and optical birefringence technique [19, 27]. The last two decades have shown that the indentation, especially nanoindentation, is a powerful method to evaluate the mechanical properties of materials. The main advantage includes the use of the same specimen in multiple tests, since nanoindentation is a non-destructive method [15]. Moreover, the theory that tensile stress is present when low resistance is the result of nanoindentation force (lower hardness values), whereas the opposite is known as compressive stress [28] have encouraged the use of this method in the study of all-ceramic materials. A disadvantage of the method is that it is only able to measure polished surfaces, i.e., flat planes [15].

The present study identified different concentrations of residual stresses of different surfaces of porcelain fused to Zirconia of 3-unit FDP with even thickness framework, especially the marginal ridges and pontic areas, based on the hardness measured by the nanoindentation method. Two hypotheses were tested: (1) the porcelain veneer located at the pontic area would present higher residual stress, and (2) the proximal marginal ridges would result in higher residual stress compared to connector marginal ridges.

2. Materials and Methods

2.1- Sample preparation

Artificial maxillary teeth, first premolar, second premolar and first molar, were positioned in a mannequin, then transferred to a 25 mm diameter PVC tube to be embedded with acrylic resin (Jet, Clássico Artigos Odontológicos, São Paulo, SP, Brazil); the enamel-dentin junction was positioned 3-mm above the acrylic resin. An impression was taken to reproduce the crown anatomy (Zetalabor, Zermack, Badia Polesine, Rovigo, Italy). Subsequently, the second premolar was removed with a diamond bur (4138 KG Sorensen, Cotia, SP, Brazil) in order to create the area for the pontic. The final dimensions of the prepared crowns were 2 mm of occlusal reduction, 1,5 mm of axial reduction and a deep chamfer margin in all surfaces of the crown. An impression was taken using polyvinyl siloxane impression material (Express – 3M Oral Care, St Paul, MN, USA) to copy the prepared teeth; later, abutment's replicas were fabricated by inserting composite resin (Z100 – 3M Oral Care, St Paul, MN, USA) though the incremental technique (2-mm increment thickness) and light-cured (Ultralux, Dabi Atlante, Ribeirão Preto, SP, Brazil) [29]. The replicas were stored in distilled water for 30 days to allow hygroscopic expansion and to eliminate possible dimensional variation after FDPs cementation [30]. Then, the abutment composite resin replicas of the first premolar and first molar were positioned into a silicone impression created previously to be embedded with acrylic resin as previously described for the intact teeth.

2.2- Y-TZP framework

The CAD-CAM system (Ceramil Amanngirbach, Koblach, Austria) was used to mill the Y-TZP frameworks from pre-sintered blocks (Ceramill ZI 71, 16mm). The acrylic resin FDP replica was positioned onto the composite abutment replicas to allow its digital scanning (Ceramill Map 400). The software could create the 3D FDP image by the acrylic resin FDP replica and the 3D prepared abutment image by the prepared artificial teeth. The image exported as .stl file allowed to use the correlation option and the abutments with framework thickness of 0.5 mm and 9 mm^2 connector area was created by reduction option. After milling, the framework was sintered in the Sintramat furnace at 1500°C for 8 h (Figure 1 - A; B; C).

2.3 - Ceramic veneering onto Y-TZP framework

The previous impression of the anatomy of the unprepared artificial mannequin teeth was used to guide the veneering ceramic hand layered application. The IPS e.Max Ceram Transpaclear (Ivoclar Vivadent AG, Schaan, Liechtenstein) veneering ceramic was applied and the firing protocol followed the manufacturer’s instructions (slow cooling and furnace opened at 450°C during glaze firing) (Figure 2). Translucent porcelain was chosen to facilitate the identification of framework and the porcelain veneer areas. The final veneering ceramic thickness was 1.5 mm on both axial walls and occlusal surface (Figure 1 - D; E).

2.4 – Cementation

Prior to the cementation procedure, the Y-TZP FDP intaglio surface was cleaned with 35% phosphoric acid (Ultra-Etch, Ultradent, South Jordan, USA) for 60s and rinsed with water for 30s. Then, the FDPs were cemented on the prepared composite resin replicas with a self-adhesive dual-cure resin cement (RelyX U200 – 3M Oral Care, St Paul, MN, USA) under a 5 Kg static occlusal load for 10 min. Subsequently, the FDPs were stored in 37^{0}C distilled water for 7 days.

2.5 – Cutting and polishing process

A 0,7 mm metallic wire was customized, then positioned and fixed with acrylate-based cement (Fisher Scientific, Waltham, MA, USA) on the occlusal surface of the FDP following the crowns central sulcus direction. Subsequently, the FDPs were embedded in epoxy resin (Resina Epóxica RD6921, Redelease, São Paulo, Brazil) and left undisturbed until completely cure for 24 hours. Then, the FDP was cut in the axial plane with a precision diamond saw (Isomet 2000, Buelher, Lake Bluff, IL, USA) under copious water irrigation. The first section was discarded, and the thicker piece containing the FDP was bonded to an acrylic plate with acrylate-based cement (Figure 1 - F; G). After a setting time of 24 h, the slide was polished (400 to 1200 grits Silicon carbide abrasive paper) 1.0 mm towards the cervical area of the FDPs, under copious water irrigation and followed by polishing (diamond suspensions of 9 to 1 µm particle size) (Buehler, Lake bluff, IL, USA). Finally, the surfaces of each FDP were inspected using a stereomicroscope (Leica Zeiss MZE, Mannheim, Germany) to check the polished surface prior to nanoindentation testing.

2.6 – Nanoindentation testing

Due the inspection under stereomicroscopy the surfaces of the specimens were divided (n=3/each) in the following groups: 1) Mesial premolar abutment (MPMa); 2) Distal premolar abutment (DPMa); 3) Buccal premolar abutment (BPMa); 4) Lingual premolar abutment (LPMa); 5) Mesial premolar pontic (MPMp); 6) Distal premolar pontic (DPMp); 7) Buccal premolar pontic (BPMp); 8) Lingual premolar pontic (LPMp); 9) Mesial molar abutment (MMa); 10) Distal molar abutment (DMa); 11) Buccal molar abutment (BMa); and 12) Lingual molar abutment (LMa).

The testing was performed at room temperature in ambient air (± 23 ºC). A nanoindenter (TI 950 TriboIndenter, Hysitron, Minneapolis, MN, USA) equipped with Berkovich diamond three-sided pyramid was used to perform the nanoindentation at porcelain veneer located at mesial, distal, buccal and lingual surfaces of the pontic and abutment crown under dry condition. The outer surface of the porcelain veneer was the reference to determine the symmetric distances between the three Regions of Interest (ROI) (ROI 1; ROI 2; ROI 3) in each surface of the crowns. The first indentation of the ROI 1, ROI 2 and ROI 3 was performed 0.03 mm, 0.35 mm and 1.05 mm from outer surface of the porcelain veneer, respectively. Each ROI received five nanoindentations with 10 µm of minimum separation and loaded to a peak load 4 µN and unloaded (Figure 3). The hardness was given by the relationship:

$$H = \frac{Pmax}{A} \qquad \text{(Equation 1)}$$

The H is the hardness value in pascal (Pa) measurement unit, Pmax is the maximum applied force in micronewton (µN) measurement unit and A is the project contact in nanomilimiter (nm) measurement unit. The values are directly obtained from nanoindentation.

2.7 – Data analysis

The Linear Mixed Model test and Least Significant Difference for multiple comparisons with mean and confidence intervals (95%) were performed using the Statistical Package for the Social Sciences (SPSS) v.18.0 (IBM Corp., USA) to compare hardness values of different surfaces of pontic and abutment crowns, specially the marginal ridges. The sum of the ROI 1, ROI 2, and ROI 3 was the total values of hardness of each surface of the 3-unit FDPs crown. Then, plots were created. Different letters on columns showed if there was any statistical difference among the groups.

3. Results

In general, the sum of all surfaces (buccal, lingual, mesial, distal) of each abutment and pontic showed that the porcelain veneer at the pontic presented the highest hardness and was statisticallly different when compared to 1st Premolar (p=0.001) and 1st molar (p=0.007). No statistical difference was observed between 1st Premolar and 1st molar (p=0.609). When buccal and lingual surfaces of abutment and pontic crowns were compared, LPMp group showed the highest hardness. Yet, this group presented statistical difference when compared to LMa (p=0.000), BMa (p=0.009), LPMa (p=0.028) and BPMp (p=0.018), whereas the BPMa group (p=0.272) did not show statistical difference (Figure 4 - A).

The comparison among marginal ridges showed that the proximal marginal ridges (DMa and MPMa) presented lower hardness values compared to connector marginal ridges (DPMa; MPMp; DPMp; and MMa). Moreover, the DMa group was statistically different when compared to DPMp (p=0.004) and MPMa (p=0.000) groups, whereas the MPMa group was statistically different when compared to DPMa, MPMp, DPMp, MMa, DMa groups (p=0.000 for all groups) (Figure 4 - B).

4. Discussion

The first postulated hypothesis that porcelain veneer located at the pontic area would present higher concentration of residual stress when compared to abutment crowns was rejected. This finding is in contrast with a previous study, which suggested that abutments (first premolar and molar) and pontic (second premolar) are not different when the porcelain veneer is hand layered using slow cooling rate for firing cycles [15]. However, the present study focused to measure the residual stress 2 mm below the occlusal plane, which allowed the nanoindentation at connector marginal ridges. This measurement was not performed in the cited study, and surprisingly this surface presented the lowest residual stress in the current study. Furthermore, the study by Baldassarri et al. used two glazing layers, which could have influenced the results.

On the other hand, it can be speculated that the higher volume of pontic crown contributed to decrease residual stress in porcelain veneer. A previous study evaluated the stress profile of the ceramic layer (1.5 mm) applied onto different framework thickness (0.5 mm, 0.7 mm, 1.00 mm, 1.50 mm, 2.00 mm, 3.00 mm); the authors concluded that the tensile stress was higher in the interior area of the porcelain veneer when thinner frameworks were used [31].

All-ceramic dental systems differ considerably from the concept of industrial glasses and ceramics, since these materials often use the rapid cooling protocol to increase their fracture resistance. The reason is related to the absence of rigid material association such as dental framework [20]. Regarding the zirconia framework, its thermal diffusivity contributes to the high presence of tensile stress from the surface of the porcelain veneer toward the framework when fast cooling protocol is used, contrasting to the cooling process of metal-ceramics restorations [15].

The second postulated hypothesis that proximal marginal ridges would result in higher concentration of residual stress compared to connector marginal ridges was accepted. Unfortunately, there is no study in literature that allows a comparison with our results. However, when premolars [11] and molars [10] human crowns are considered, the proximal marginal ridge results of the present study corroborate to previous studies which suggest that the marginal ridges and proximal areas present the highest tensile stress. A photoelastic previous study of porcelain veneered fused to zirconia crowns showed that curved areas present higher concentration of residual stress regard of slow and fast cooling protocols [19, 20], which is in line with a recent experimental and finite element study [18] that found higher stresses in curved interfaces after evaluating the residual thermal stress of bars, semi-cylindrical shells, and arch-cubic structures bilayered with 1.5 mm and 0.7 mm thickness of porcelain veneer and zirconia framework, respectively using the slow cooling at 32 °C/min and extremely-slow cooling at 2 °C/min protocols.

In the present study, the method could inspect the most significant stress of the porcelain veneer fused to zirconia of 3-unit FDP, which is located closer to the superficial occlusal area of porcelain veneer [18, 31, 32]. In addition, the study evaluated anatomically-relevant FDPs, which differ from studies that used geometric shapes, such as: flat layers [31, 32], bars, shell or arch-cubic [18] and virtually specimens for finite element analyses (FEA) studies [13, 33, 34].

The FEA studies have reported different scenarios of porcelain veneer fused to zirconia FDPs. In general, most of the studies show that the highest tensile of stress occur at the basal side the connector. When 3-unit and 4-unit FDPs were analyzed, the gingival embrasure of the connectors between premolars / molars and the middle connector [13, 33] present the maximum of tensile stress, respectively. However, only one point of static loading located at the center of the FDPs is very often used, which can influence significantly the stress induced on FDPs. Some authors have suggested the distribution of occlusal contacts to minimize this discrepancy effects for simulating usual clinical loading conditions [33, 34].

A recent 3 years follow up study [9] of 4- to 6-unit porcelain veneer fused to zirconia FDPs (tooth and implant retained restorations) described the chippings location on abutment and pontic crowns. For tooth-retained restorations the total of 5 chippings were found around FDPs and could be divided into: 4 chipping at pontic crowns and only 1 fracture at abutment crown. Most of the time the chippings at pontic crown involved the occlusal, followed by lingual, buccal and mesial surfaces. For implant-retained restorations, the total of 9 chippings were found and divided into: 5 chippings at abutment crown and 4 chippings at pontic crowns. Most of the chipping of abutment involved the mesial followed by lingual, occlusal and buccal surfaces, whereas the pontic crowns involved the lingual, followed by mesial, occlusal and buccal surfaces. The polishing was used to repair all the chippings, regardless of the retained restorations.

Clinical follow-up studies have shown that connector fractures of Y-TZP frameworks are rare thus far, regardless of location at posterior or anterior region [1-4, 35]. Therefore, studies involving porcelain veneer of prostheses should be more present in literature.

5. Conclusion

The concentration of residual stress at the porcelain veneer located on pontic and abutments were different, which may explain clinically observed failure locations. The connector marginal ridges presented the lowest residual stress values.

ACKNOWLEDGEMENTS

This study was supported by Fundação de Amparo à Pesquisa do Estado de São Paulo (São Paulo Research Foundation - FAPESP) grants: 2011/16377-0, 2011/23177-8, 2013/20806-0, 2014/22859-6, 2012/19078-7 - Young Investigators Awards, and **EMU 2016/18818-8.** Also in part by Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) grant # 309475/2014-7 and 309475/2014-7.No manufacturer supported this study.

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Captions to Figures

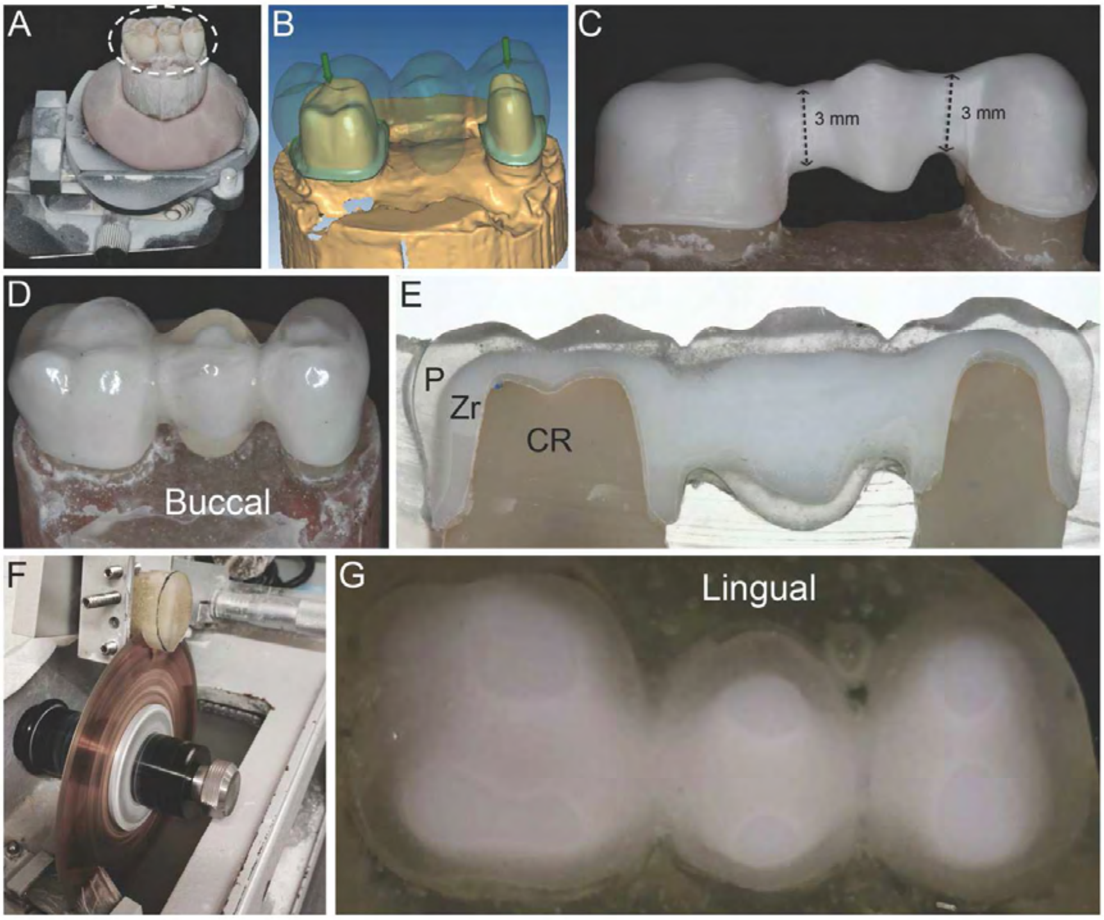

Figure 1 - (A) Cad/Cam spray used onto the acrylic resin replica (circle) to increase the contrast before scanning. (B) The 3D FDP positioned onto the 3D abutments using the correlation option. (C) Y-TZP framework after sintered in the Sintramat furnace. (D) Hand layered translucent porcelain after glazing. (E) Area distribution of porcelain veneer (P), zirconia (Zr) and composite resin (CR). (F) Diamond saw used to apply the second cut of the FDP parallel to the metallic wire. (G) Approximate view of the specimen after cutting.

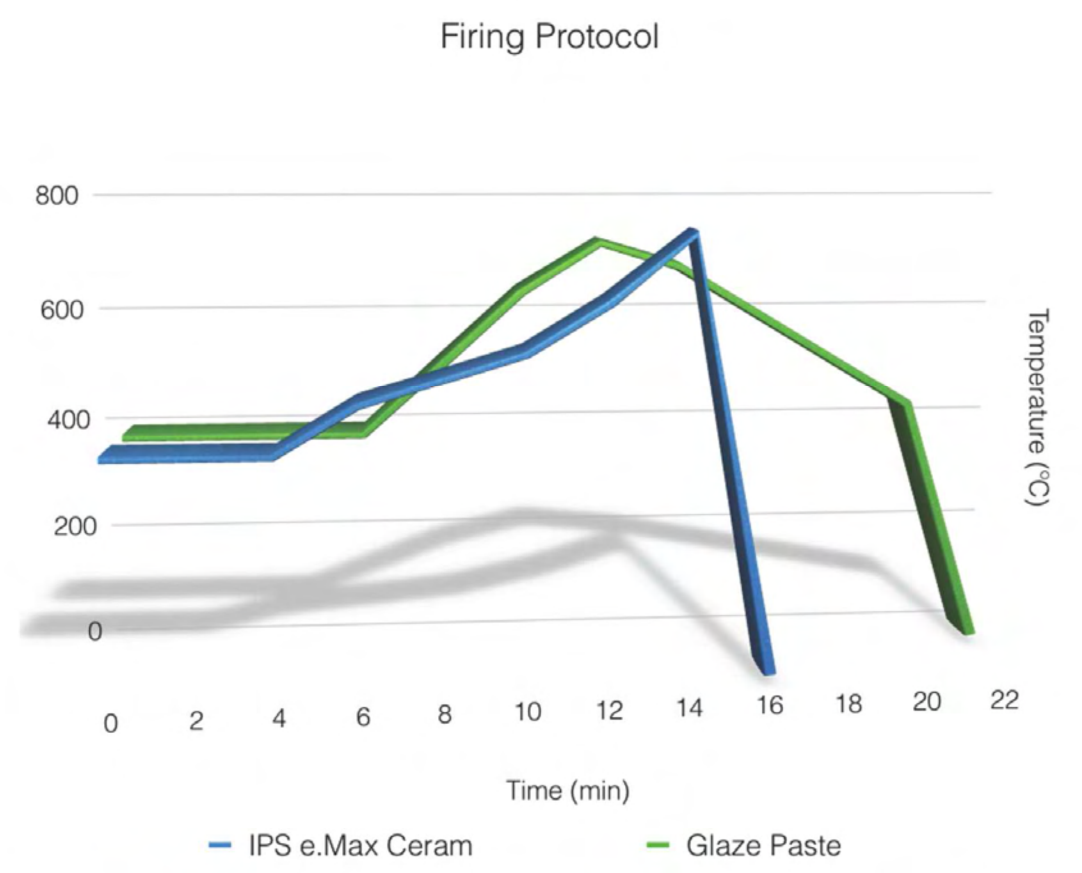

Figure 2 – The slow cooling protocol for Glaze Paste was used following the manufacturer recommendation.

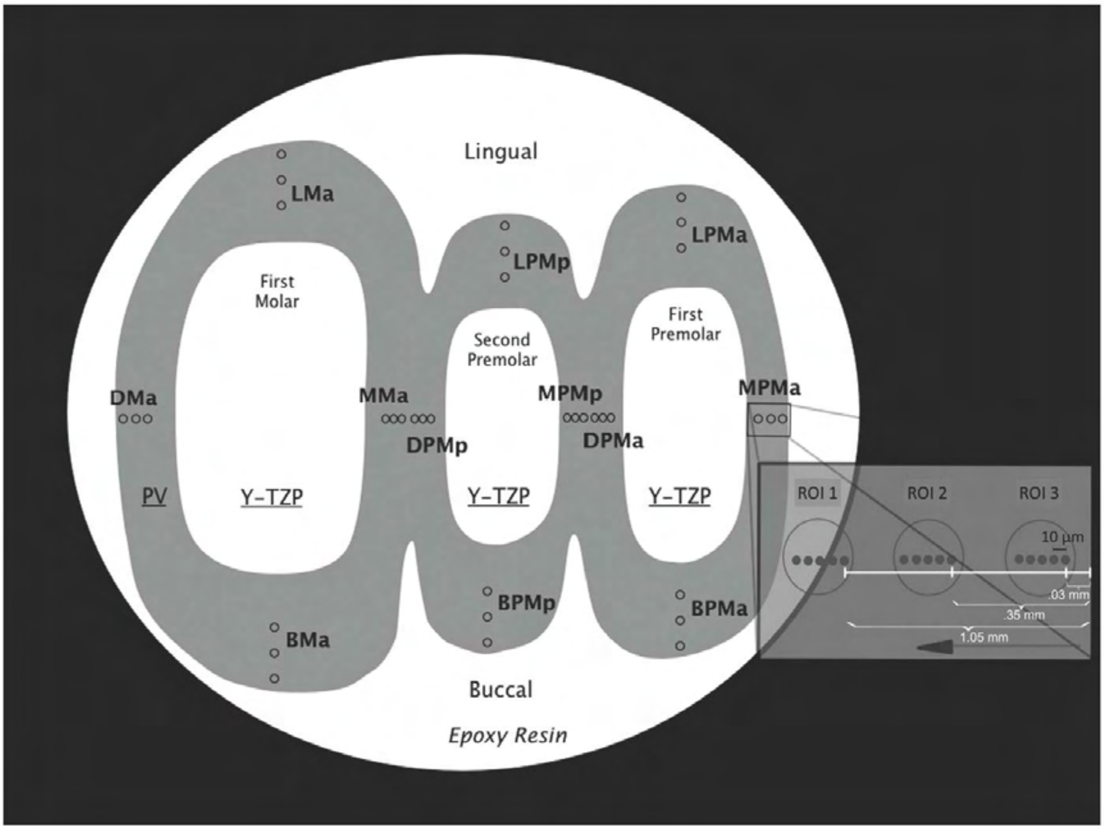

Figure 3- Schematic view showing the group locations, zirconia framework (Y-TZP), porcelain veneer (PV), abutments (First Molar and First Premolar) and pontic (Second Premolar) of the specimen. The magnified view showed the distances nanoindented, which were 0.03 mm, 0.35 mm and 1.05 mm from outer porcelain surface for ROI 1, ROI 2, and ROI 3, respectively. The ROI 3 was consistently the closest ROI to the porcelain veneer/framework interface. Five nanoindentation points with 10 µm of minimum separation was performed in each spot to provide a more precise hardness value and the arrow demonstrated the sequence of the nanoindentation test.

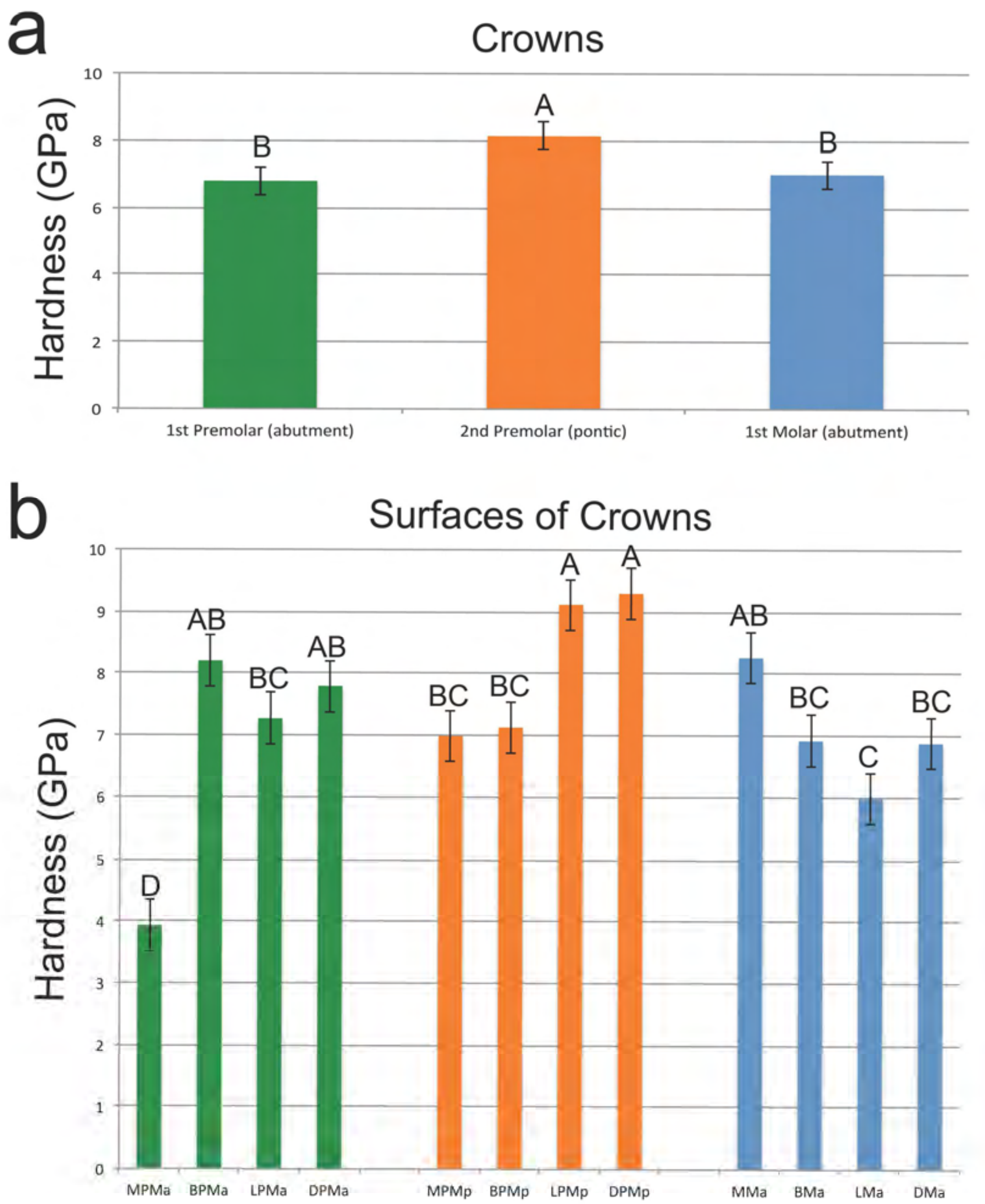

Fig. 4 - Statistical difference was found for the plots presenting different letters. (a) Higher hardness value was found in pontic crown. (b) Lower hardness values were found in proximal marginal ridges, when only marginal ridges are compared.

3 Discussion

3 DISCUSSION

This study focused on different concentrations of residual stress found in different surfaces and regions of interest (away and close to the PV/framework interface) of fatigued and non-fatigue porcelain veneer fused to zirconia 3-unit FDPs with different frameworks designs. The nanoindentation method was used to measure the hardness values of 3-unit FDPs where high or low values corresponded to low and high concentration of residual stress, respectively. The main findings of the present study were: a) the outer surface of the porcelain veneer (ROI 1) presented the highest concentration of residual stress and was significantly different when compared to ROI 2 and ROI 3, regardless of nanoindentation occurring at abutments or pontics; b) the residual stress was significantly higher in fractured compared to suspended fatigued specimens from the same framework design groups (3.5 times more residual stresses) c) the porcelain veneer at the pontic and, mainly, the connector marginal ridges of ZrEven group (non-fatigued FDPs) presented the lowest concentration of residual stress.

The first hypothesis that postulated that fractured and suspended samples would not show different residual stresses regardless of the framework design was rejected. The comparison between ZrEvenF and ZrEvenS showed that the residual stress increased more than 2.3 times in ZrEvenF group. Also, a similar finding of residual stress was observed between the comparison of ZrModF and ZrModS groups, where the former showed an increase over 3.5 times in residual stress compared to the latter. Unfortunately the present study results could not be compared with any data in literature.

The framework design suggested for porcelain veneered Y-TZP FDPs was based on the fact that the enlargement of the connector height (33) and the gain of framework volume, even based on empirical guidelines for metal ceramics (34) would decrease the high concentration of residual stress of FDPs, specially in connector areas, which is the location that presents the highest concentration of stress (35-37). In this way, its use would provide the decrease porcelain fracture size, the inhibition of gingival connector cracks and higher strength in connector areas (38). However, the present study showed that 3-unit ZrMod groups revealed to be efficient only in inhibiting the cracks located at gingival connector areas. Even so, out of forty couple of fatigued marginal ridges, only one specimen from Y-TZP even thickness framework presented this type of damage, where competitive crack development is observed between indentation sites and the gingival embrasure areas. These findings highlight the controversial benefits of the frameworks with different designs when different loading location, different slow cooling protocols and fatigue methods are performed on crowns (29, 39-42).

However, it seems that the human factor may influence the different concentration of residual stress even applying the slow cooling protocol followed by manufacturer recommendation. It is well known that the conventional manual layering technique may create structural flaws located at framework / porcelain veneer interface, in the bulk of the veneer layer, or at the surface of the porcelain veneer (43). This technique allows the incorporation of voids, impurities and porosities in the porcelain veneer, which may create sites with high tension of residual stress even fabricating restorations in a controlled environment with a vacuum furnace (43-48). The veneering techniques (49) and multi-layer structure (50) have a crucial influence on the behavior of bilayered prostheses (48) and consequently its stress distribution may become more complex than expected. Another critical factor that can increase the residual stress is associated to spending overtime of layering the framework using inadequate proportion of powders and pastes. On the other hand, a study (51) that evaluated porcelain veneered crowns using the press technique as an alternative for conventional manual layering showed that the porcelain fractures were also present. Moreover, no statistical difference was found when the technique was compared to crowns with porcelain layered by manual technique, The reason was correlated to the structural flaws that could not be avoided with the press veneered technique, concluding that porcelain is a friable material.

The second hypothesis postulating that the layer closer to the glazing surface would present higher stress compared to the layer closer to the framework/porcelain veneer interface was accepted. This result corroborated with a recent study (23) that evaluated the residual stresses in bars, semi-cylindrical shells and arch-cubic structures fabricated with different cooling rates. The different distances measured from the framework / porcelain veneer interface in parallel directions were 0.4, 0.8, and 1.3 mm showed that the compressive stress became higher closer to the interface in parallel distances (23). Another nanoindentation study showed the geometric influence of crowns on measuring the residual stress regardless of the thickness of the porcelain veneer (9), where the maximum stress values occurred, in general, over curved surfaces of the framework (20). To avoid potential inconsistencies, the sectioning height of the specimens used in this study showed to be safe in the standardization of the distances from framework / porcelain veneer interface (ROI 1, 2, and 3). Besides nanoindentation studies, this study is in agreement with the findings from the hole-drilling method (52) that measured the stress profile in bilayered discs presenting different thickness of framework and porcelain veneer. Its result showed that compressive stresses were found in the veneering ceramic surface of thicker frameworks and tensile stress were observed in the interior of thinner framework samples (52). However, when different thickness of veneering ceramic were evaluated, the compressive stress located at the ceramic surface for samples veneered with 1.0 mm decreased from the surface at 0.5 mm until 0.7 mm and became more compressive again close to the framework (52). Yet, that study showed that crack propagation tended to follow tensile stress areas, thus higher tensile stress was commonly found closer to outer porcelain veneer surface, which may explain the reduced sizes of porcelain veneer fractures (52).

The benefits of the lower residual stress present in ROI closer to the framework can be explored when the classification of porcelain fracture sizes is considered. Chipping (small porcelain cohesive fractures) was the most common fracture and the fracture dimensions were smaller than 2 x 2 mm, except for one larger fracture was found in ZrEvenF group. However, all the chippings were repairable by composite resin or returned to function by polishing. Currently, the literature described that there is a tendency to find minor chipping in follow-up clinical studies (3, 5) as long as the manufacturer's slow cooling protocol recommendation is followed (5). Laboratory studies have shown improvements in performance when long slow cooling protocol is used (8, 42).

The third postulated hypothesis described that porcelain veneer located at the pontic area would present higher residual stress was rejected. This finding is not in agreement with a previous study, which suggests that abutment (first premolar and molar) and pontic (second premolar) are not different when the porcelain veneer is hand layered using slow cooling rate for firing cycles (14). However, the present study focused on measuring the residual stress of 2 mm below the occlusal plane, which allowed the nanoindentation at connector marginal ridges. This measurement was not evaluated in the cited study, and surprisingly this surface presented the lowest residual stress in the present study. Furthermore, the previous study used two glazing layers, which could influence the final results.

On the other hand, it is suspected that pontic areas presents higher volume of Y-TZP, which can decrease the presence of residual stress in porcelain veneer. A previous study measured the stress profile in bilayered disc samples with different framework thickness (0.5 mm, 0.7 mm, 1.00 mm, 1.50 mm, 2.00 mm, 3.00 mm) and with a 1.5 mm thick veneering ceramic layer with hole-drilling method showed that the tensile stress became higher for interior area of the porcelain veneer when the thickness of the framework decreased (52).

The fourth postulated hypothesis described that the proximal marginal ridges would result in higher residual stress compared to connector marginal ridges was accepted. Unfortunately, there is no study in literature that allows a comparison between the results. However, when premolars (24) and molars (25) human crowns are considered, the marginal ridges and proximal areas presented the highest tensile stress which corroborate with the present study. In addition, a photoelastic previous study of porcelain veneered fused to zirconia crowns showed that curved areas present higher concentration of residual stress regardless of slow or fast cooling protocols (16, 20). This result is in agreement with a finite element study (23) that performed the slow cooling at 32 °C/min and extremely-slow cooling at 2 °C/min protocols and found higher stresses in curved interfaces of bars, semi-cylindrical shells, and arch-cubic structures bilayered with 1.5 mm and 0.7 mm thickness of porcelain veneer and zirconia framework, respectively.

The present study assumed high or low concentration of residual stress based only on hardness values as previously reported by Zhang et al (9), whereas other studies have presented different calculations to determine the residual stress within the porcelain veneer (16, 23, 53). In such scenarios, baseline samples in unstressed conditions are commonly subjected to indentations allowing specific equations to be used for this purpose (53). Since our study involved anatomically-shaped FDPs, laboratory processing commonly results in samples with residual stress of either tensile or compressive nature (54). While we were unable to produce unstressed samples, control non-fatigued FDPs were used instead. In silico analysis has also been used not only in crown-shaped samples (16) but also on bar shaped samples (23). Additional studies should be targeted to determine the type of residual stress, (compressive or tensile) within regions of the porcelain veneer in anatomically-relevant specimens.

4 Conclusions

4 CONCLUSIONS

Based on the studies described in this thesis, we conclude that:

1. The redisual stress of porcelain veneer of fractured or suspended fatigued Y-TZP 3-unit fixed dental prostheses with the same framework designs presented different concentration of residual stress.

2. The outer layer of the porcelain veneer presented the highest concentration of residual stress in abutments and pontic.

3. The concentration of residual stress found in abutments was higher than in pontics.

4. The connector marginal ridges presented the highest hardness values.

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Annexes

Annex 1

Submission letter to the *Journal of the Mechanical Behavior of Biomedical Materials*.

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Research Paper

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