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Fracture Load and Phase Transformation of Monolithic Zirconia Crowns Submitted to Different Aging Protocols

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Clinical Relevance

Advances in zirconia that have resulted in favorable esthetic and mechanical properties have enabled its application as a full-contour restoration.

SUMMARY

Monolithic zirconia crowns have many favorable properties and may potentially be used to solve dental problems such as chipping. However, monolithic zirconia crown resistance can be affected by its phase transformation when subjected to low temperatures, humidity, and stress. This study evaluated the fracture load and phase transformation of monolithic zirconia crowns submitted to different thermal and mechanical aging tests. Seventy monolithic zirconia crowns were randomly divided into the following five groups: control, no treatment; hydrothermal aging at 122°C, two bar for one hour; thermal fatigue, 10^4 cycles between 5°C and 55°C, dwell time, 30 seconds; and mechanical fatigue, 10^6 cycles with a load of 70 N, sliding of 1.5 mm at 1.4 Hz; and combination of mechanical plus thermal fatigue. Fracture load was measured with a universal testing machine. Surface changes and fracture mode and origin were examined with a scanning electron microscope. Monoclinic phase content was evaluated by x-ray diffraction. The fracture load was analyzed using one-way analysis of variance at a level of 5%, and Weibull distribution was performed. No statistically significant differences were observed in the mean fracture load and characteristic fracture load among the groups ($p>0.05$). The Weibull modulus ranged from 6.2 to 16.6. The failure mode was similar for all groups with the crack origin located at the contact point of the indenter. Phase trans-

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formation was shown at different surfaces of the crown in all groups (1.9% to 8.9%). In conclusion, monolithic zirconia crowns possess high fracture load, structural reliability, and low phase transformation.

INTRODUCTION

In the early 1990s, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was proposed for use in dentistry, and interest in this material has grown due to its superior mechanical properties compared with other ceramic systems.[1,2] The excellent mechanical behavior of Y-TZP is credited to a transformation toughening mechanism, in which tetragonal zirconia crystals transform into the monoclinic form and produce compressive stresses that prevent cracks and hinder crack propagation.[2-4] Due to the relatively low fracture rate of Y-TZP structures, a wide range of applications with high clinical success rates has been proposed for these materials, such as dental prostheses and implant abutments.[5-7]

Prosthetic dental crowns are conventionally fabricated in two steps: construction of a framework and subsequent veneering with an esthetic veneering ceramic. When Y-TZP prostheses were first proposed for dental applications, the prostheses were veneered with compatible porcelain to improve the final esthetic result. Nevertheless, this type of prosthesis has demonstrated relatively high clinical fracture rates, usually expressed as chipping and/or delamination of the porcelain layer.[8,9] To overcome this problem, manufacturers have recently proposed the use of Y-TZP as a monolithic (full-contour) restoration (ie, without the veneering layer). The production of monolithic dental crowns only became possible due to the development of modified Y-TZP microstructures with higher translucency and the addition of coloring pigments.[10] This type of restoration achieves reasonable esthetic results via the addition of a superficial glaze layer that allows for final biomimetic characterization. The increasing popularity of monolithic Y-TZP crowns for prosthetic rehabilitation is partially related to economic issues. Specifically, production of these crowns requires fewer manufacturing steps and less human labor because they are almost entirely processed via Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) systems.[11-13] Additional advantages of monolithic Y-TZP crowns have been reported in the literature, including reduced tooth preparation[12-14] and superior fracture resistance compared with other ceramic materials.[11,13,15]

When using monolithic Y-TZP crowns, the zirconia surface may be directly exposed to adverse oral conditions, and in some cases, the surface is only protected by a thin glaze layer. Low temperature degradation (LTD) may be triggered when zirconia surfaces are in direct contact with water at body temperature.[16,17] This type of degradation occurs through energy barrier reduction for tetragonal to monoclinic transformation caused by the incorporation of water into the zirconia lattice.[18-20] LTD can occur at low temperatures (range = 37°C to 500°C), with a maximum transformation rate occurring at 250°C.[16,21] This phenomenon is time dependent and proceeds gradually from the surface into the bulk of the ceramic by a nucleation-and-growth process characterized by surface roughening, microcracking, and macrocracking, which enables water to deeply penetrate the material.[16,21-23] Therefore, LTD progression can significantly affect Y-TZP mechanical properties.[24,25]

In addition to LTD, zirconia-based materials undergo fatigue and subcritical crack growth under functional loading in the oral environment, which gradually reduces their strength.[26,27] Continuous contact with teeth during mastication can damage the material surface and cause the accumulation of critical defects, which may lead to catastrophic failure over time. [26,28] However, zirconia monolithic crowns have been proven to cause lower antagonist wear than other materials, such as lithium disilicate and dental porcelains.[11] In addition to mechanical degradation due to chewing, repeated thermal variations in the oral cavity also generate tensile stresses within the zirconia crown, accelerating the fatigue process via subcritical crack growth.[28,29] A previous study reported a significant reduction in the load-bearing capacity of zirconia framework prostheses after combined mechanical and thermal cycling.[27] However, whether the observed degradation was caused by LTD or mechanical fatigue remains unknown.

Due to the increasing use of zirconia as a monolithic restoration material and the evidence that its mechanical properties can be negatively affected by phase transformation triggered by superficial stresses or LTD,[23-26,30-34] this study evaluated the effect of different hydrothermal and mechanical aging processes on the fracture load and phase transformation of monolithic zirconia crowns. Our null hypothesis asserted that different aging procedures would not affect the mechanical behavior and the monoclinic phases of Y-TZP crowns.

METHODS AND MATERIALS

Specimen Preparation

A three-dimensional (3D) CAD model of a left maxillary first molar was generated. To simulate the preparation of conventional all-ceramic prostheses, the tooth was anatomically modeled by reducing the proximal walls by 1.5 mm and the occlusal surface by 2.0 mm, with a convergence angle of 10 degrees between the mesial and distal walls as well as between the buccal and palatal walls. Furthermore, a marginal chamfer preparation of 1.0 mm was employed.[35]

A new commercially available highly translucent yttria-stabilized zirconia (Ceramill Zolid, Amann Girrbach, Koblach, Austria), which has a grain size ≤0.6 μm, was utilized. Based on a 3D model previously developed, the monolithic zirconia crown was designed and milled with the CAD/CAM system Ceramill Motion 2 (Amann Girrbach) derived from white presintered blocks (n = 70). After the milling procedure, the crowns were stained with Ceramill coloring liquids (Amann Girrbach) following the manufacturer's guidelines. Then, the samples were sintered at 1,450°C for two hours and glazed according to the manufacturer's instructions (Ceramill stain and glaze, Amann Girrbach).

A CAD-prepared tooth model was machined in an acrylic resin block (VIPI blocks, VIPI, Pirassununga, Brazil). Seventy prepared tooth replicas were fabricated from polyvinylsiloxane impressions (Futura, Nova DFL, Taquara, Brazil) of the plastic model. The impressions were filled with layers of resin-based composite (Z250, 3M/ESPE, Sumaré, Brazil) and light cured according to the manufacturer's recommendation. Then, each replica was embedded in acrylic resin (Dencôr, Clássico, São Paulo, Brazil) into a 15-mm-diameter polyvinyl chloride tube (PVC), leaving 1 mm exposed from the buccal margin preparation. The replicas were stored in deionized water at 37°C for 30 days to ensure complete hydration of the samples and to eliminate any dimensional expansion effect due to water absorption.

The Y-TZP crowns were then cemented with dual-curing resin composite cement (Panavia F 2.0 - LOT 000003; Kuraray, Tokyo, Japan) and ultrasonically cleaned. The resin replicas were etched with phosphoric acid for five seconds, and a ceramic primer was applied according to the manufacturer's instructions. Then, the ED Primer II mixture was applied in the replicas for 30 seconds after which the resin composite cement was mixed, applied in the crown, and inserted in the tooth replica. The crowns were maintained under a load of 10 N and the excess removed. The margin was light-cured for 20 seconds on each surface. After cementation, the specimens were stored in deionized water for seven days to provide suitable aqueous equilibrium prior to testing.[36]

Aging Procedures

The crowns were randomly divided into five groups (n = 14) that received the following aging treatments corresponding to a lifetime of approximately one year *in vivo*.[23,37,38]

1. Control: no aging treatment.
2. Hydrothermal aging: aging was carried out in a reactor controller (Reactor Parr 4843, Parr, Moline, IL, USA), a chamber that controls temperature and pressure, in this case, at 122°C (±1°C) under two bars for one hour.[23]
3. Thermal fatigue: specimens were submitted to 10^4 thermal cycles[37] in distilled water between 5°C and 55°C, with a dwell time of 30 seconds.
4. Mechanical fatigue: samples were mounted in a PVC matrix filled with acrylic resin (Dencôr) and placed in a chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany). Each specimen was subjected to 10^6 mechanical cycles[38] with a load of 70 N applied by sliding a stainless steel antagonist (5.6 mm in diameter)[13] through a 1.5-mm path at the inner side of the mesiopalatal cusp from the lingual to the buccal side at a 1.4-Hz frequency. The crowns were immersed in distilled water at 37°C during mechanical cycling. All specimens were evaluated for the presence of damage and cracks at the end of the cycling period.
5. Combination of mechanical and thermal fatigue: samples were sequentially submitted to the mechanical and then to the thermal fatigue protocol.

Fracture Load Measurement

At the end of each aging treatment, 12 crowns belonging to each group were loaded until a fracture occurred using a universal testing machine (Kratos KE, Kratos Equipment, Cotia, Brazil) equipped with a load cell of 10 KN at a crosshead-speed of 1 mm/min. The force was applied at the central fossa of the occlusal surface via a stainless steel ball (5.6 mm in diameter),[13] and the crowns were immersed in distilled water at 37°C during the test. Load at

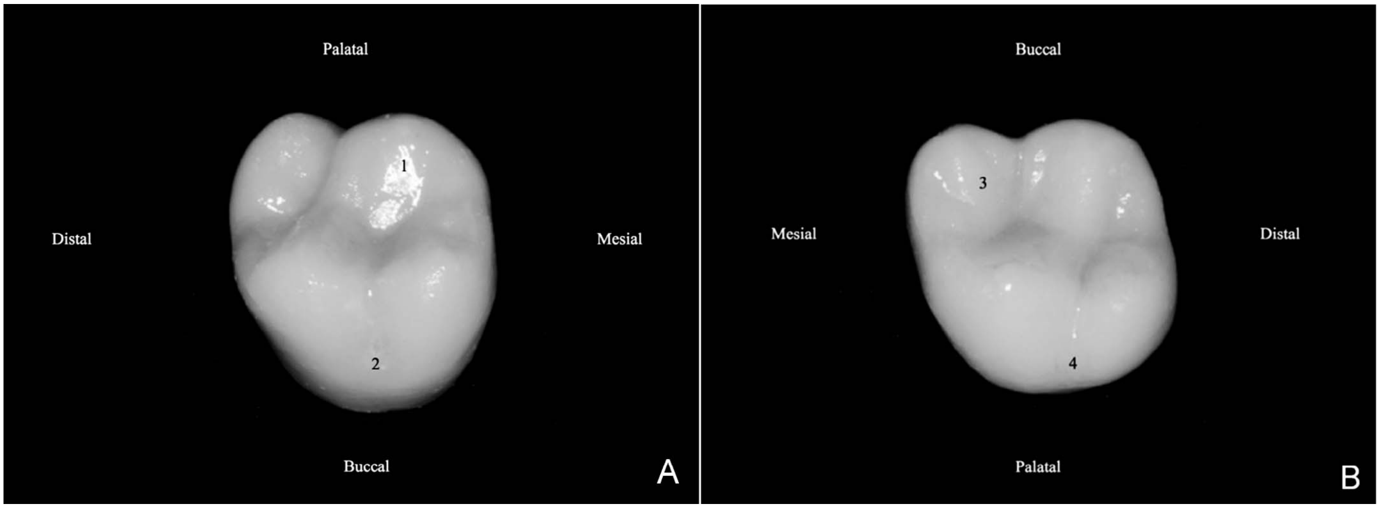

Figure 1. *Points selected to perform X-ray diffraction analysis: (A): 1, mesiopalatal cusp; 2, buccal surface. (B): 3, mesiobuccal cusp; 4, palatal surface.*

fracture (N) was registered, and a fracture was defined as a visible fracture or as the occurrence of an acoustic event and load drop.

Phase Transformation

The relative amount of monoclinic phase after aging procedures was determined by x-ray diffraction in the palatal and buccal surfaces and the mesiobuccal and mesiopalatal cusps of two samples from each group (Figure 1). The x-ray diffraction profiles were obtained using a diffractometer dispensing energy at 8 keV (Cyberstar detector, FMB Oxford, Oxford, United Kingdom). The x-ray tube is stationary, the specimen displaces at an angle of θ, and the detector simultaneously moves at an angle of 2θ. To evaluate the microstructural characterization of the crowns, scans were performed at 2θ, ranging from 20 to 70 degrees with incremental increases of 0.05 degree. The monoclinic peak intensity ratio (Xm) was calculated using the following equation[39]: Xm = $I_{m(-111)} + I_{m(111)} / I_{m(-111)} + I_{m(111)} + I_{t(101)}$ where I_t and I_m represent the integrated intensity area under the peaks of the tetragonal (101), monoclinic (−111), and monoclinic (111) peaks at 30°, 28°, and 31.2°, respectively. The data obtained from this equation were used to calculate the monoclinic volume content at the surface (Vm)[40] : Vm = 1.311.Xm / 1 + 0.311.Xm

Scanning Electron Microscopy

To evaluate the surface modifications after aging, crowns from each group were sputter coated with gold/palladium alloy (Bal-Tec 020, Leica Microsystems, Wetzlar, Germany) for scanning electron microscopy (SEM) analysis (Jeol JSM-6360LV; Jeol, Boston, MA, USA). After fracturing, the specimens were examined with a light-polarized stereomicroscope (Olympus SZ61, Olympus, Waltham, MA, USA), and representative specimens were evaluated by SEM to identify the fracture origin and to characterize the fracture mode.

Statistical Analysis

Normal data distribution was confirmed by the Shapiro-Wilk test. Mean fracture loads of the groups were analyzed by one-way analysis of variance. Statistical calculations were performed using the SAS system release 9.3 (SAS Institute Inc, Cary, NC, USA), and a significance level of 5% (α=0.05) was adopted. The reliability of the Y-TZP crowns was assessed through the Weibull distribution (Weibull++, ReliaSoft, Tucson, AZ, USA), and the analysis was performed to determine the Weibull modulus and the characteristic failure load in each group. The Weibull modulus determines the slope of the distribution function and characterizes the spread of the data with respect to fracture load. Characteristic failure load (P_0) defines the stress level at which 63.21% of the specimens fail.[41,42] The Weibull distribution parameters were estimated by the maximum likelihood method, and their 95% confidence intervals (CIs) were computed. Differences among groups were considered to be significant when the 95% CIs did not overlap.

RESULTS

All restorations survived the artificial aging tests, and no damage was visible to the naked eye. Although a

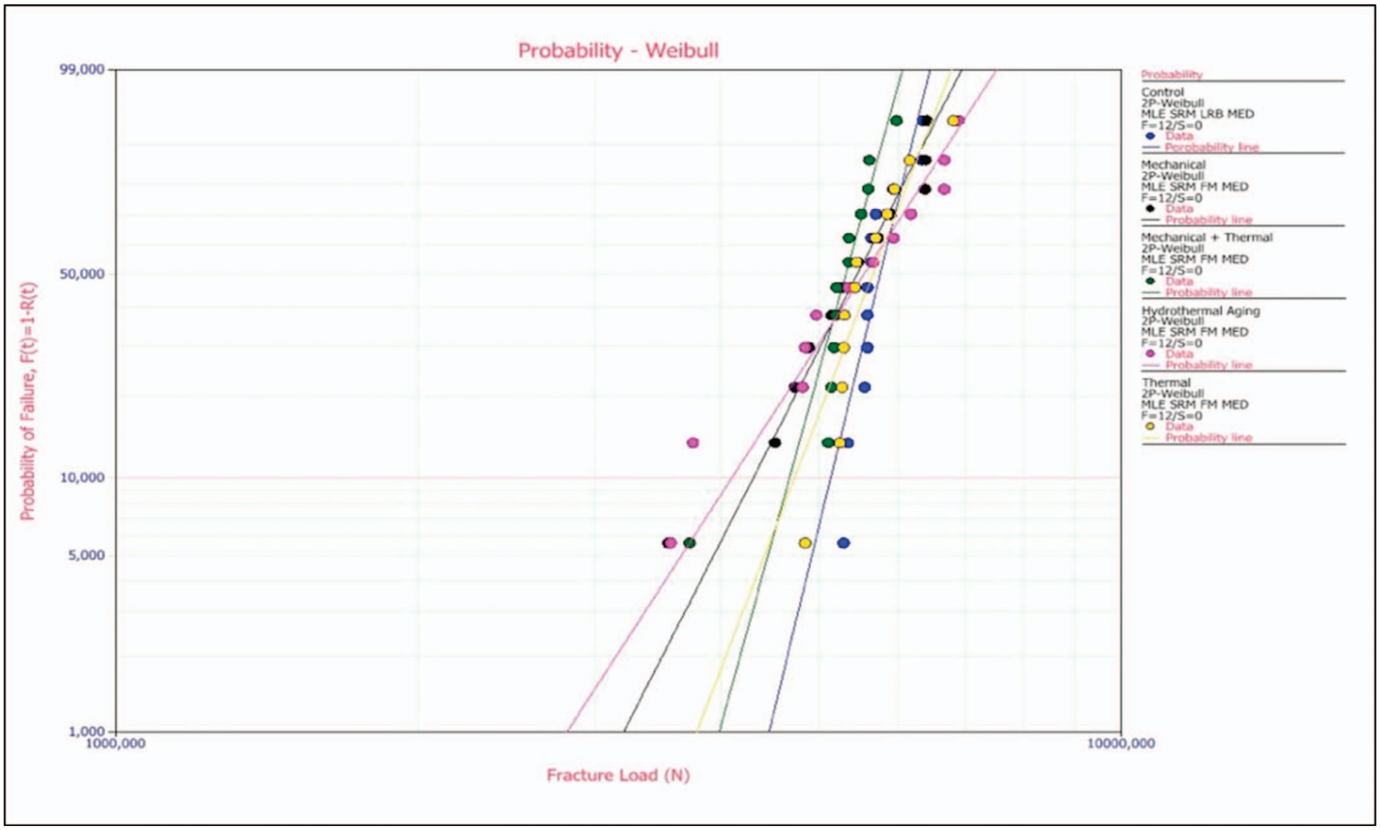

Figure 2. *Curves representing the Weibull probability of fracture for each group (confidence interval of 95%).*

decrease in the mean fracture load was observed for all age groups in comparison to the control, no statistically significant difference was observed for any of the pairwise comparisons ($p>0.05$). Similar findings occurred for the characteristic fracture load (P_0) as the Weibull statistics did not detect a significant effect of aging (Figure 2; Table 1).

The Weibull modulus ranged from 6.2 to 16.6 (Table 1). The Weibull modulus values of the age groups decreased compared with the control; however, the decrease in the Weibull modulus in relation to the control was only statistically significant for the group submitted to hydrothermal aging (Figure 2; Table 1).

The x-ray diffraction analysis indicated that the control group contained a specific level of monoclinic phase (approximately 4%) in most of the evaluated surfaces. Hydrothermal and thermal aging protocols did not alter the amount of monoclinic phase in the measured spots. For groups submitted to mechanical aging (mechanical fatigue and mechanical plus thermal fatigue), the amount of monoclinic phase increased more than eightfold on the mesiopalatal cusp, where attrition with the steel ball occurred (Figures 3 and 4).

The SEM micrographs of representative occlusal surfaces showed significant glaze layer degradation

Table 1: *Fracture Load and Weibull Distribution Parameters of Groups, Characteristic Failure Load (P_0), and Weibull Modulus (m) (Confidence Interval Limits of 95%)[a]*

Group	Mean Fracture Load (N)	Weibull Parameters	
		Characteristic Failure Load (P_0) (N)	Weibull Modulus (m)
Control	5722 (5934-5510)[a]	5884 (5652-6109)[a]	16.6 (10.4-24.0)[a]
Hydrothermal aging	5450 (6149-4750)[a]	5867 (5342-6467)[a]	6.2 (3.9-9.9)[b]
Thermal	5618 (5949-5287)[a]	5858 (5532-6204)[a]	10.5 (7.0-15.6)[a,b]
Mechanical	5538 (5997-5079)[a]	5718 (5304-6164)[a]	7.9 (5.0-12.5)[a,b]
Mechanical + thermal	5256 (5602-4911)[a]	5455 (5239-5679)[a]	14.6 (9.3-22.7)[a,b]

[a] *Different letters in each column represent significant difference among the groups ($p<0.05$).*

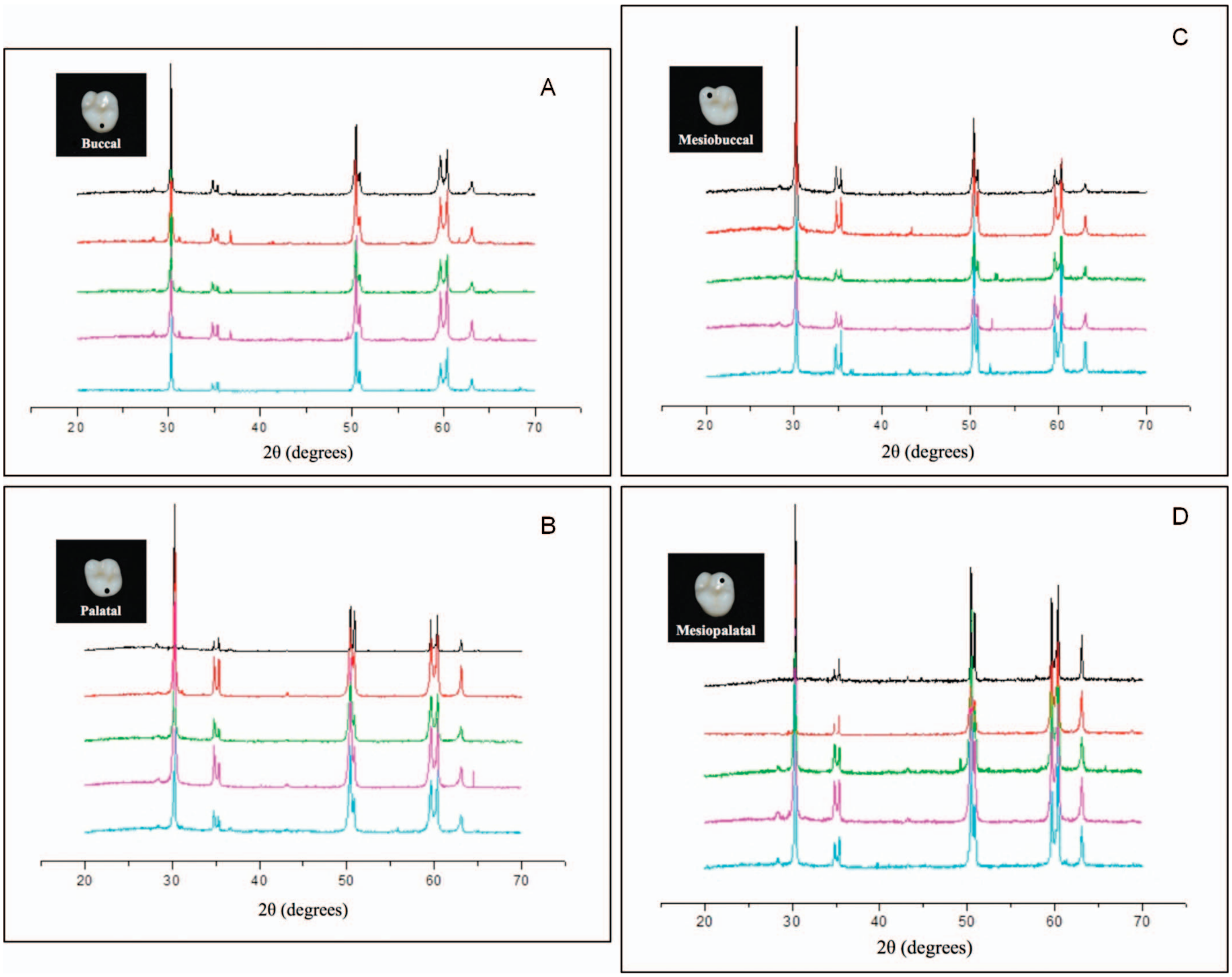

Figure 3. *Diffractogram of crowns according to tooth surface and aging treatment. Crown surfaces: (A): buccal; (B): palatal; (C): mesiobuccal cusp; and (D): mesiopalatal cusp. Aging treatment: black, control; red, hydrothermal aging; green, thermal; pink, mechanical; blue, mechanical and thermal fatigue.*

in the groups subjected to mechanical aging (mechanical fatigue and mechanical plus thermal fatigue), with marked damage accumulation and exposure of the underlying Y-TZP (Figure 5). The failure modes were similar for all experimental groups, with the crack origin located at the contact point of the indenter. The central fossa and the main crack propagated from the occlusal surface toward the crown cementation surfaces (Figure 6).

DISCUSSION

This study evaluated the fracture load and phase transformation of monolithic zirconia crowns submitted to different thermal and mechanical aging tests on prosthetic crowns that were designed to mimic clinical situations. The primary hypothesis was only partially accepted because although the aging protocol did not affect the fracture load of the Y-TZP monolithic crowns, the amount of monoclinic phase at the crown surface was higher for protocols using mechanical cycling.

All tested crowns resisted the applied artificial aging methods, and no statistically significant difference was observed among the mean fracture loads and characteristic fracture loads of all experimental groups. The absence of significant differences among the fracture loads obtained with the aging methods could be due to weak parameters incapable of degrading the mechanical behavior of the tested Y-TZP monolithic crowns in the aging protocols.

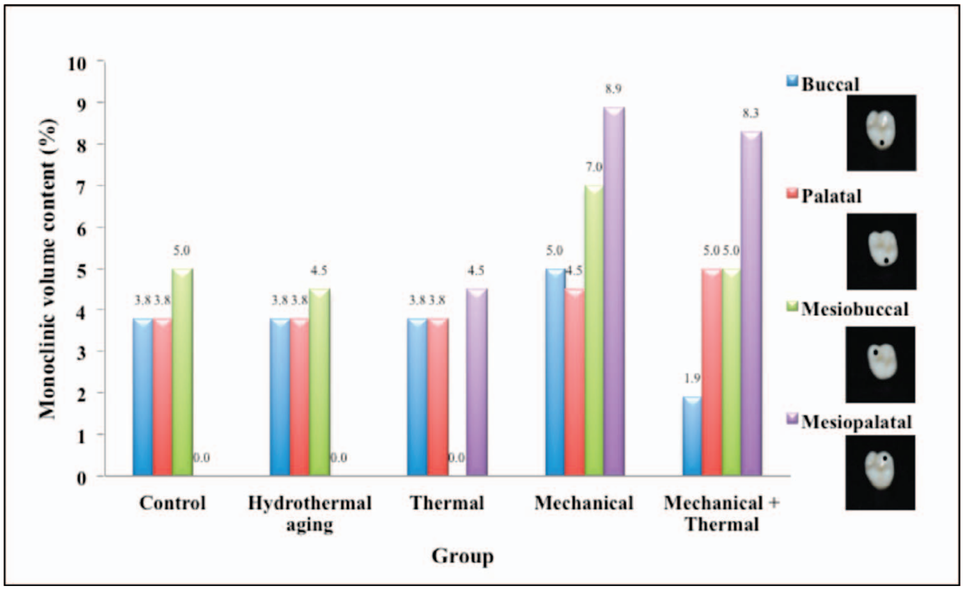

Figure 4. *Relative amount of monoclinic phase detected by x-ray diffraction in each aging group at different crown surfaces.*

Regarding mechanical cycling, the load chosen for the aging protocol (70 N) may have been insufficient to generate a stress level along the Y-TZP crown that could significantly degrade its mechanical properties. Importantly, Y-TZP has a significant phase transformation strengthening mechanism that hinders slow crack propagation[2-4,25] and increases the lifetime of the prosthetic crown by protecting the crown from adverse effects of the oral cavity environment.

The fracture load values obtained in the present study (5,256-5,722 N) far exceeded the load levels found clinically regarding the maximum bite force (900 N).[43] Few reports have evaluated the fracture load of monolithic zirconia crowns, and previously published results are considerably different from the results found in the current experiment. One previously published study reported fracture load values of up to 10,000 N,[11] while others obtained lower values varying from 2,795 to 3,068 N.[13,15] A recently published study showed a load-bearing capacity of 5,620 N for monolithic zirconia crowns, which is closer to the values identified in the present study.[14] In different studies, the large variation observed in the fracture load values for the same type of prosthetic crown (monolithic Y-TZP) can be explained by significant experimental differences among these works, such as the direction and location of the load in relation to the crown surface as well as the type of abutment substrate, indenter characteristics (eg, material type and dimensions), prosthetic crown thickness,[44] presence or absence of a wet environment during the test,[15] and previous aging treatments before the fracture test.[11,15] Other factors may also affect the measured fracture load of Y-TZP crowns such as differences in the number of layers applied during the glaze procedure and the total number of firing cycles conducted for glaze layer sintering.

The Weibull modulus is the shape parameter of the Weibull distribution and is used to describe the variation in strength values as a result of the type of flaw population present in the material structure. Higher Weibull modulus values correspond to a more homogeneous flaw size distribution, less data scatter, and greater structural reliability. Conversely, the opposite is expected from lower Weibull modulus values.[41,42] This study showed Weibull modulus values ranging from 6.2 to 16.6, which is consistent with the values reported for ceramic crowns.[45] As per the Weibull modulus values shown in Table 1, only hydrothermal aging appeared capable of causing a significant decrease in Y-TZP crown reliability. Although no surface phase transformation was detected in the x-ray diffraction analysis for specimens submitted to hydrothermal aging, further structural analyses such as micro-Raman and x-ray photoelectron spectroscopy are needed to investigate whether the occurrence of nonhomogeneous phase transformation at the crown surface and within the crown may be responsible for the significant drop in the reliability level of this experimental group.

Regarding the fracture modes, all specimens showed catastrophic failures with the crack origin located at the occlusal surface at the loading point between the indenter and the central fossa. From this point, the crack propagated toward the intaglio

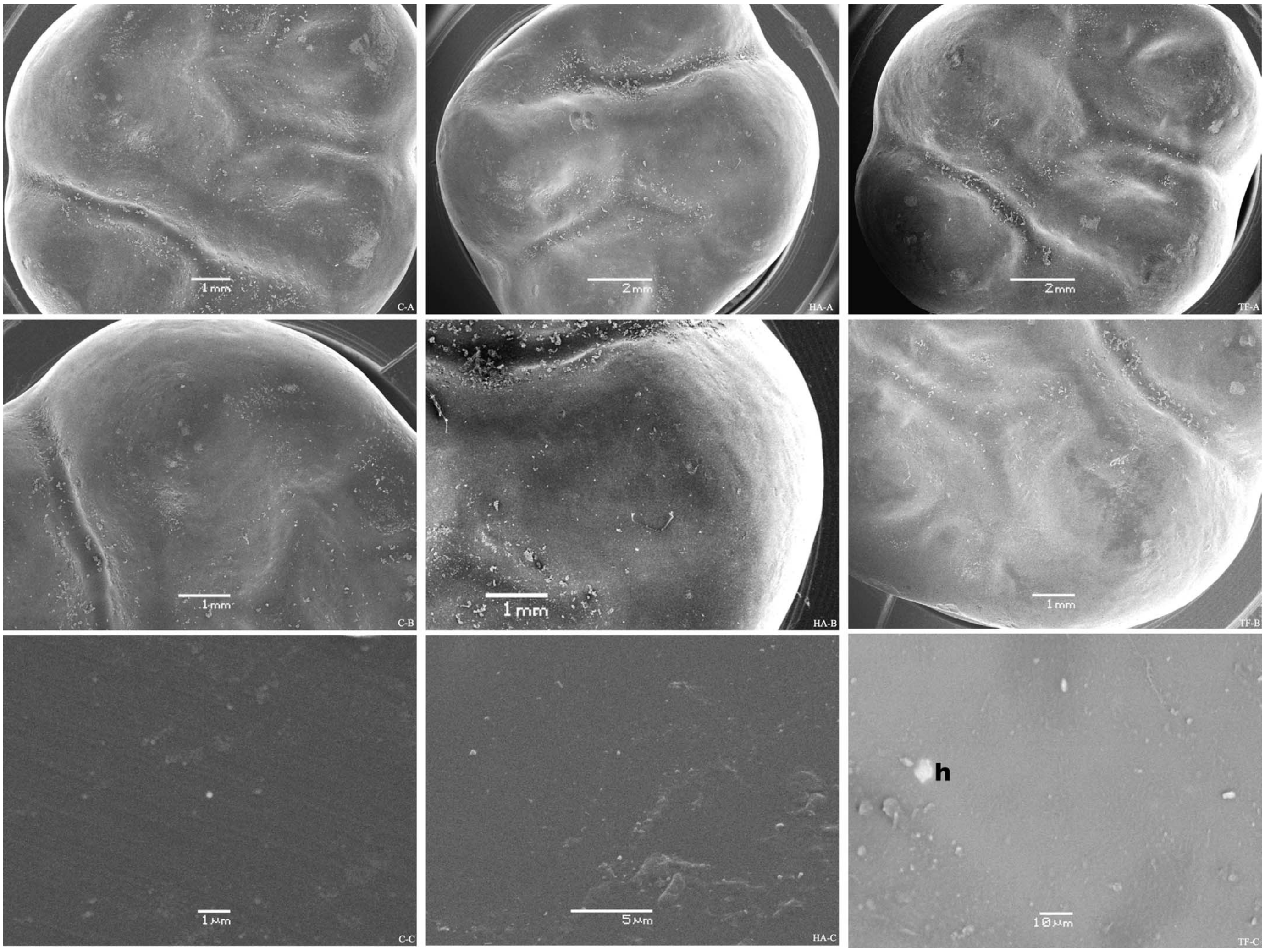

Figure 5. *Column A shows the surface view of zirconia monolithic crowns after aging treatments: C, control; HA, hydrothermal aging; TF, thermal; MF, mechanical fatigue; and MTF, mechanical and thermal fatigue. Column B depicts mesiopalatal cusp views of different groups, where MF and MTF exhibit the loss of glaze layer and zirconia exposure due to mechanical stress. Column C shows a closer view of the integrity of the glaze layer in C and HA; a hole is shown in the TF group; and the interface between the glaze layer (g) and zirconia exposure (z) (arrow) is shown in the MF and MTF groups.*

surface of the crown. This type of fracture mode is typically observed in the fracture surfaces of monolithic crowns in laboratory studies that used methodologies similar to the one used in this investigation.[13,15,28] Catastrophic failures of monolithic Y-TZP crowns occur at much higher stress levels compared with those needed to fracture the veneering porcelain layer applied over Y-TZP copings; therefore, the fracture mode observed in the present investigation indicates an important mechanical advantage for the full-contour crowns[13]. Indeed, *in vitro* studies that determined the fracture strength of zirconia crowns veneered with porcelain reported mean fracture loads varying from 1480 to 2500 N.[13,15] These values are considerably lower than the fracture load found in the current investigation for monolithic zirconia crowns (approximately 5200 N).

With respect to phase transformation, a certain amount of monoclinic phase content was already noted in the control group (Vm = 0 to 5%). The presence of a monoclinic phase in Y-TZP surfaces submitted to a glaze layer was expected because previous work has proven that the application of a wet porcelain slurry to Y-TZP surfaces with subsequent firing results in a localized tetragonal to monoclinic transformation in the core/veneer interface.[33,34] The x-ray diffraction analysis was capable of detecting transformations that occurred at the interface of the Y-TZP crown with the applied thin glaze layer.

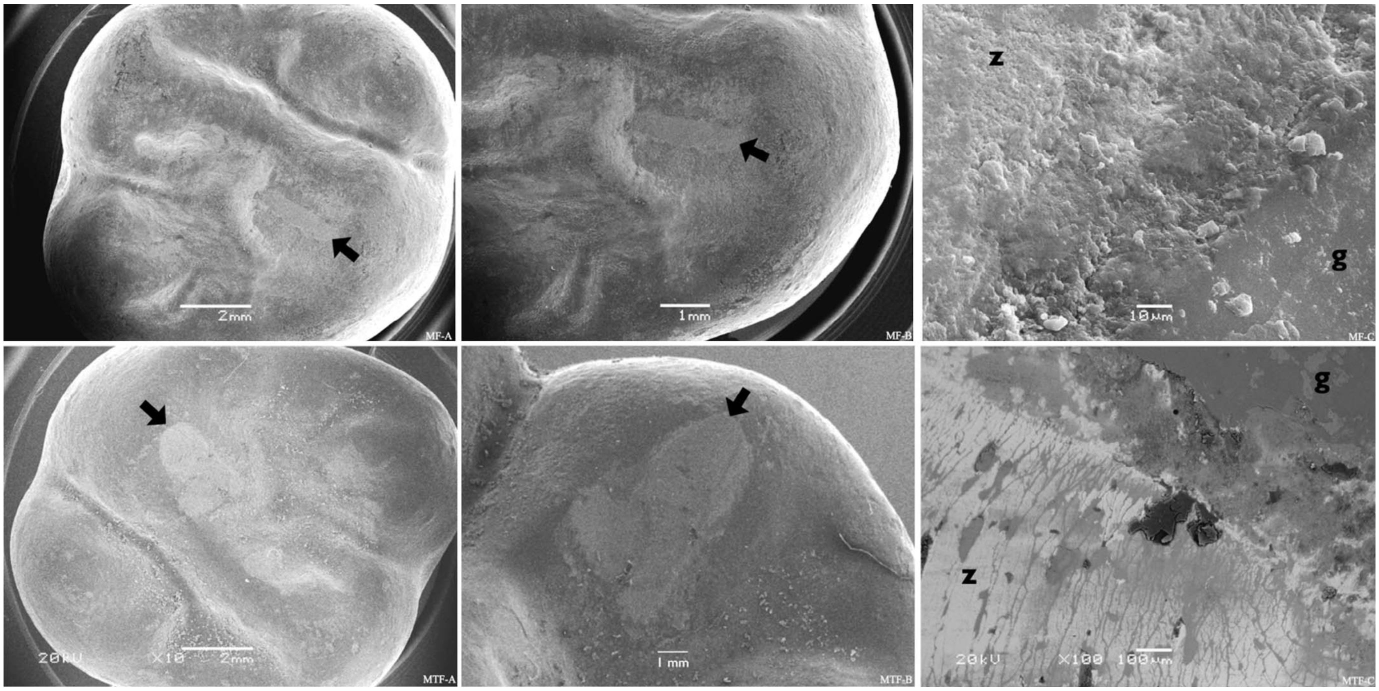

Figure 5. *Continued.*

Material strength is degraded by repeated thermal stresses in prosthetic restorations. These thermal stresses lead to tensions within the specimens and manifest in slow subcritical crack growth and catastrophic failure.[27] Nevertheless, in the present study, thermal fatigue and hydrothermal aging resulted in similar monoclinic volume content compared with the control at all surfaces (Vm = 0 to 4.5%). This result can be attributed to the presence of the glaze layer that protects the zirconia surface from water contact and avoids additional phase transformation. A previous study revealed phase transformation (Vm = 6.4%) after 3×10^4 thermal cycles,[31] and another study showed no monoclinic phase after two hours of hydrothermal aging in the same conditions used in the present investigation[30]; however, both studies evaluated Y-TZP specimens without a glaze layer, confirming the hypothesis of the protection offered by the glaze layer or insufficient aging proposed by the current investigation.

The mechanical fatigue and mechanical plus thermal fatigue aging groups showed a slight increase in the monoclinic volume content (Vm = 8.3 to 8.9%), especially in the mesiopalatal cusp, in comparison to the same surface of the other groups (Vm = 0 to 4%). This increase in monoclinic content after aging is most likely related to the attrition and mechanical stresses generated by the contact of the crown surface with the indenter. Indeed, the surface SEM micrographs of the crowns showed significant damage of the glaze layer with exposure of the underlying Y-TZP surface after the chewing simulation. A previous study[32] that investigated the phase transformation caused by mechanical stresses in zirconia blocks revealed higher monoclinic volume content (Vm = 13%) in comparison to the current experiment. This difference is also most likely related to the distinct conditions of the two studies, such as load level, number of cycles, and presence of the glaze layer.

The diffraction peaks obtained by x-ray diffraction need to be analyzed with caution because although this technique is considered the first step to investigate zirconia aging sensitivity, no precise information can be obtained during the initial transformation stages (Vm values < 5%), and significant variability of the results may occur when different locations on the same specimen are analyzed.[46] The x-ray beam can only interact with the outermost surface of the material with a penetration depth of a few micrometers[46]; therefore, the presence of the thin glaze layer may have affected the test precision.

Considering the results of the present investigation, further laboratory studies need to be conducted to better comprehend the mechanical behavior of monolithic zirconia crowns. Future work should use cyclic fatigue methodologies that will determine the

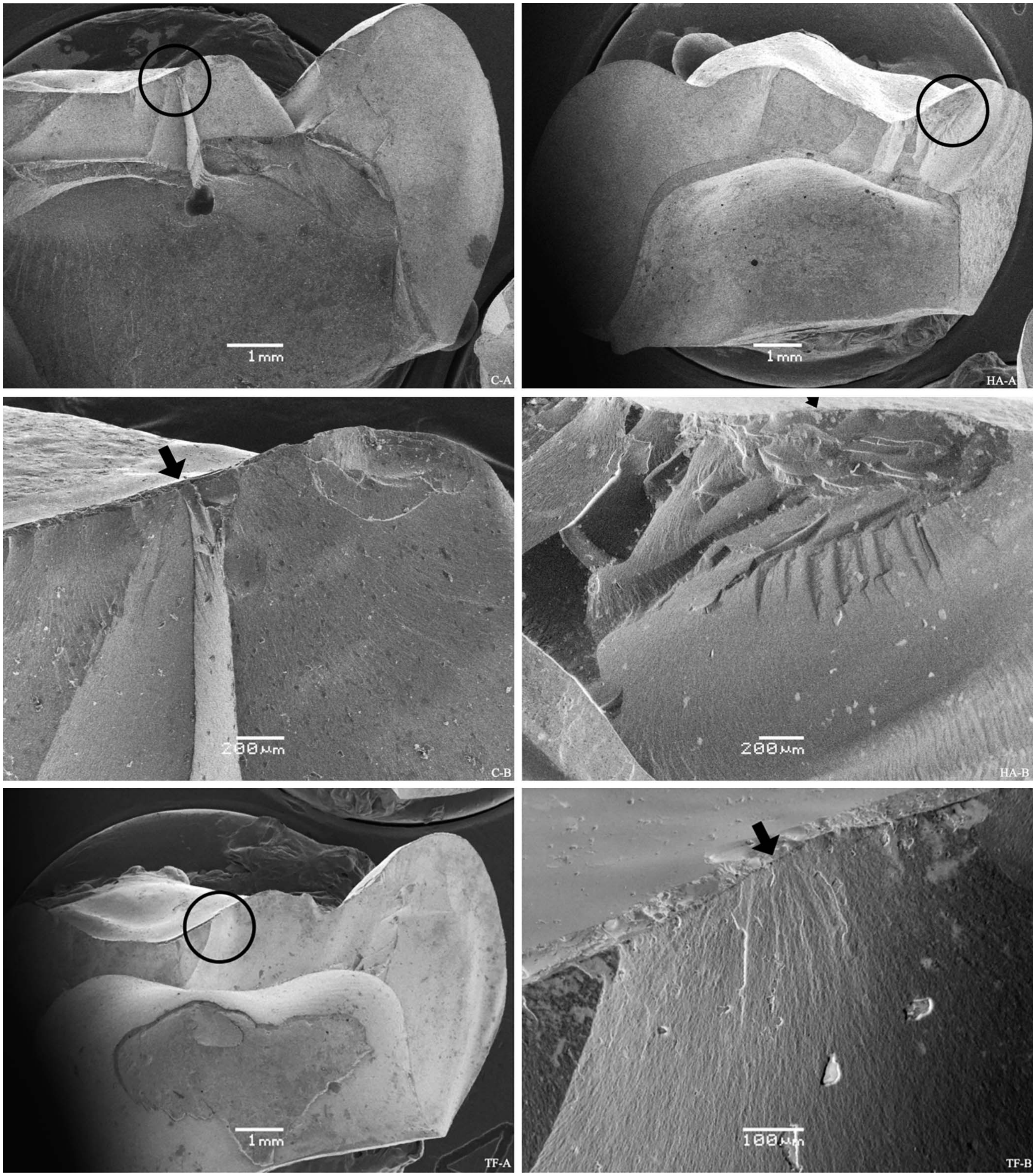

Figure 6. *Fractographic analysis. All groups showed the same failure mode with the crack origin (circle and arrow) located at the surface in contact with the indenter. Column A shows no approximated views and column B shows closer views of the groups: C, control; HA, hydrothermal aging; TF, thermal; MF, mechanical fatigue; and MTF, mechanical and thermal fatigue.*

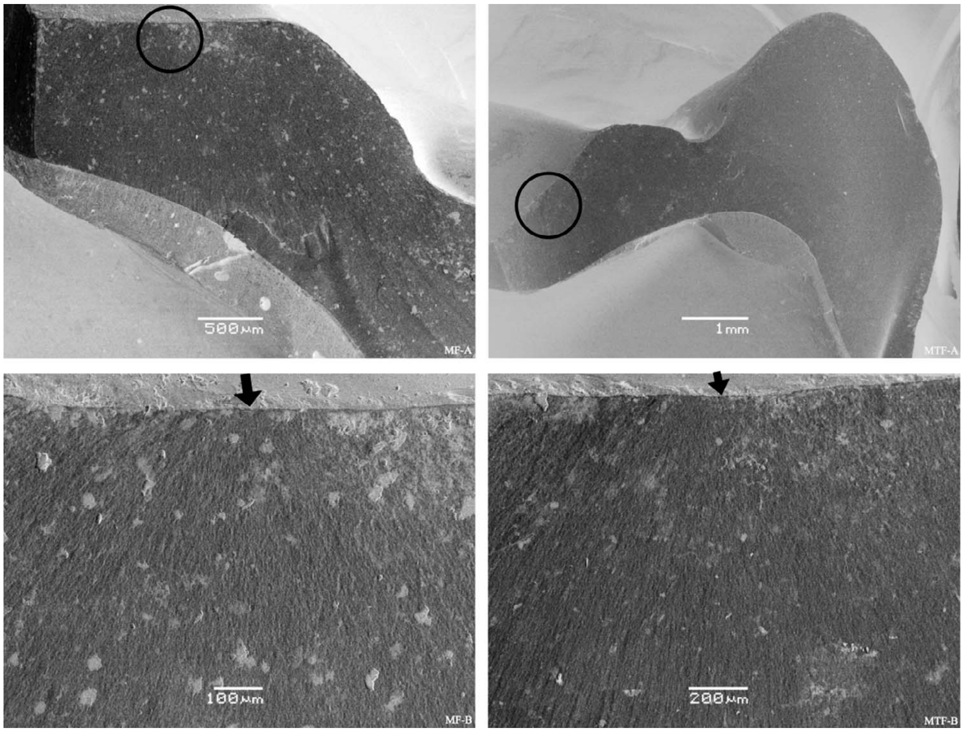

Figure 6. *Continued.*

material lifetime and fatigue parameters. Also, longer aging protocols should be tested to clarify how superficial phase transformation occurs over longer clinical lifetimes.

CONCLUSION

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

- None of the four different aging protocols affected the fracture load of the tested Y-TZP monolithic crowns;
- Only the hydrothermal method reduced the structural reliability of the crowns;
- The aging protocols involving attrition of an antagonist with the Y-TZP glazed layer were the only protocols resulting in a significant increase in the monoclinic phase at the crown surface;
- The SEM observations indicated accumulated damage of the glaze layer and subsequent exposure of the zirconia surface due to attrition with the antagonist during the chewing simulation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Piracicaba Dental School, University of Campinas.

Conflict of Interest

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

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