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RESEARCH AND EDUCATION

Effects of artificial aging conditions on yttria-stabilized zirconia implant abutments

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ABSTRACT

Statement of problem. Most ceramic abutments are fabricated from yttria-stabilized tetragonal zirconia (Y-TZP). However, Y-TZP undergoes hydrothermal degradation, a process that is not well understood.

Purpose. The purpose of this in vitro study was to assess the effects of artificial aging conditions on the fracture load, phase stability, and surface microstructure of a Y-TZP abutment.

Material and methods. Thirty-two prefabricated Y-TZP abutments were screwed and tightened down to external hexagon implants and divided into 4 groups (n = 8): C, control; MC, mechanical cycling (1×10^6 cycles; 10 Hz); AUT, autoclaving (134°C; 5 hours; 0.2 MPa); and TC, thermal cycling (10^4 cycles; 5°/55°C). A single-load-to-fracture test was performed at a crosshead speed of 0.5 mm/min to assess the assembly's resistance to fracture (ISO Norm 14801). X-ray diffraction (XRD) analysis was applied to observe and quantify the tetragonal-monoclinic (*t-m*) phase transformation. Representative abutments were examined with high-resolution scanning electron microscopy (SEM) to observe the surface characteristics of the abutments. Load-to-fracture test results (N) were compared by ANOVA and Tukey test (α=.05).

Results. XRD measurements revealed the monoclinic phase in some abutments after each aging condition. All the aging conditions reduced the fracture load significantly (P<.001). Mechanical cycling reduced the fracture load more than autoclaving (P=.034). No differences were found in the process of surface degradation among the groups; however, the SEM detected grinding-induced surface flaws and microcracks.

Conclusions. The resistance to fracture and the phase stability of Y-TZP implant abutments were susceptible to hydrothermal and mechanical conditions. The surface microstructure of Y-TZP abutments did not change after aging conditions. (J Prosthet Dent 2016;116:277-285)

The introduction of zirconia-based ceramics as a dental material has been gaining considerable interest within the dental community. Since the mid-1970s, much research has been done on the subject as a result of the discovery of the phase transformation toughening of zirconia.[1] Their mechanical properties are reportedly the strongest of all ceramic materials.[2]

At room temperature and pressure, the monoclinic (*m*) phase is the stable structure of pure zirconia. Alloying zirconia with stabilizing oxides can keep the tetragonal (*t*) crystallographic structure at room temperature.[3] In response to an external mechanical stress, a structural *t-m* phase transformation may take place at crack tips. The resulting volume expansion leads to compressive stresses in opposition to crack propagation, thereby enhancing the fracture toughness, which, in turn, may also result in higher strength.[1,4,5] Therefore, the use of zirconia, in particular, yttria-stabilized tetragonal zirconia (Y-TZP), has extended the application of ceramic

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Clinical Implications

The assembly's decreased resistance to fracture was not only the result of hydrothermal degradation. The effects of mechanical fatigue may result in a more significant clinical representation than steam autoclaving.

restorations to ceramic abutments where strength and esthetics are essential.

Implant-supported, single-tooth restorations are subjected to the most precise requirements in esthetic locations.[6] Success depends on proper osseointegration, optimal implant positioning, functional load capability in the long term, and satisfactory esthetic outcomes. Despite the improvements in the design of transmucosal titanium abutments, their dark shade may negatively affect the appearance of periimplant tissues and impair the esthetic results.[7,8]

In the search for a ceramic abutment to solve the shortcomings of titanium, Y-TZP was introduced as a candidate material. Among ceramic abutments, in vitro studies[9-11] have confirmed the superior fracture resistance of Y-TZP abutments, which have been able to withstand high loads of up to 1016 N.[12] Accordingly, a prospective clinical study of Y-TZP abutments supporting single-tooth crowns exhibited excellent long-term outcomes, with a cumulative success rate of 96.3% after 11 years of use.[13] However, such predictions must be moderated by the relatively short observation time in mostly prospective studies. Indeed, clinical reports of catastrophic failures do exist.[14]

One critical drawback of ceramic materials is their susceptibility to fatigue mechanisms that can considerably reduce their strength and, therefore, the lifetime of such components.[15-17] Particularly, because Y-TZP continues to be in a metastable state, a slow *t-m* transformation may enhance its susceptibility to fracture.[18] This process, also called low-temperature degradation (LTD), is a well-described phenomenon. Nucleation, which corresponds to the *t-m* transformation of 1 or a few surface grains, leads to a volume increase that causes stress to the neighboring grains that may trigger the propagation from one transformed grain to the others. The growth of transformed zones is related to extensive microcracks offering a path for water to penetrate into the bulk of the material. Thus, LTD propagates by a nucleation and growth process that may significantly reduce the mechanical properties.[19-24] Some types of zirconia are susceptible to aging, and manufacturing conditions can play a critical role in the LTD process.[22,25-29]

Although the LTD in zirconia materials was studied in detail on flat specimens, few attempts have been made to study aging in complex geometries.[27,28] The aging process in terms of structural integrity must be defined for the future use of Y-TZP as an implant abutment material. Currently, a lack of correlation exists between aging and clinical failures, mainly because there is no uniform approach to conducting scientific studies. Therefore, evaluating aging in an in vitro setting that reflects a more significant clinical representation appears to be useful. The purpose of this study was to evaluate the effect of different artificial aging conditions on the fracture resistance, *t-m* phase transformation, and surface microstructure of a Y-TZP implant abutment.

MATERIAL AND METHODS

Thirty-two 1-piece Y-TZP prefabricated abutments (Implant Neodent Osseointegrável) on external hexagon implants with regular 3.75-mm diameter and 13-mm length (Titamax Ti Cortical; Implant Neodent Osseointegrável) were used in this study. Implants were embedded in epoxy resin (Araldite GY1109; Huntsman Química Brasil Ltd), which was polymerized for 4 hours at 70°C. A metallic device was used as a guide to allow each implant to be embedded up to a distance of 3 mm below the nominal level specified by the implant manufacturer to simulate bone resorption. This procedure ensured that all implants were placed concentrically and at a standardized embedment depth.[30]

To prepare the assemblies, abutments were attached to the implants by titanium alloy retention screws (Implant Neodent Osseointegrável) at the manufacturer's recommended torque (32 Ncm) using a calibrated digital torque gauge (Instrutherm TQ-680; Instrutherm Measuring Instruments Ltd). After 10 minutes, the screws were retightened to prevent preload loss.[31] The implant-abutment assemblies were divided into 4 groups and subjected to 1 of the following aging conditions (n = 8): C, no further treatment (control); MC, mechanical aging in water (1×10^6 cycles, 10 Hz, at 37°C); AUT, accelerated aging test for 5 hours in autoclave; and TC, thermal cycling (1×10^4 cycles between 5°C and 55°C).

Subsequently, the MC test group underwent fatigue testing following ISO 14801, as this standard simulates the functional loading of an endosseous dental implant body and its prosthetic components in a critical in vivo scenario.[30] A custom-designed steel holder was made to fix the specimens in a universal testing machine (model 8872; Instron Corp) at 30 ±2 degrees with respect to the vertical axis. According to ISO 14801, the loading center should be at the longitudinal distance of 11 mm from the support level of the implant.[30] To standardize the distance, a metal hemispherical cap with the matching shape of the abutment was fabricated and temporarily cemented (Provy; Dentsply Intl) on each abutment, ensuring that the loads were applied with the same lever arm. Based on the assembly's performance in previous studies, a cyclic loading between 11 and 211 N was chosen.[32,33] The test was performed in distilled water at a frequency of 10 Hz and an upper limit of 1×10^6 cycles.[34-36]

The AUT test group was subjected to an accelerated aging test in an autoclave at 134°C under 0.2 MPa pressure for a 5-hour run.[37] One hour at 134°C would roughly correspond to 2 years at 37°C.[38] The effects of aging were evaluated after 5 hours because this aging period represents the expected lifetime of zirconia restorations (5 hours corresponds to 10 to 20 years in vivo).[28]

The TC test group was aged with a thermal fatigue simulator device (MSCT-3; Elquip) by means of 1×10^4 thermal cycles in distilled water between 5°C and 55°C (30-second dwell time at each temperature).[39]

Monoclinic content was analyzed on flat surfaces at the lingual aspect of all abutments (control and test groups) with an x-ray diffraction (XRD) technique (CuKα 1.5418 Å, 40 kV, and 30 mA) with an x-ray diffractometer (D5000; Siemens, Bruker AXS). Diffraction data were first collected in a continuous mode, $\Delta2\theta$ = 4 to 70 degrees, with a step size of 0.05 degrees and a scan speed of 1 degree/s. To allow for quantitative measurement of the observed monoclinic phase, the abutments that presented monoclinic content were then evaluated in a continuous mode, $\Delta2\theta$ = 20 to 70 degrees, with a step size of 0.02 degree and a scan speed of 10 degrees/s.

XRD data and the Rietveld method were used for the quantitative phase analysis.[40,41] The background was refined using software (TOPAS academic v5.0; Coelho Software), which was corrected by Chebyschev polynomial function using 5 terms. The following parameters were refined: atomic coordinates, occupancies, unit cell, scale factor, specimen displacement, and atomic displacement. In all abutments, monoclinic Inorganic Crystal Structure Database (ICSD) 82543 and tetragonal ICSD 89429 were identified.[42,43]

After XRD analysis, control and test groups were subjected to a single-load-to-fracture test by using a 30-degree angled steel holder in a universal Material Testing System 801 (MTS Systems Corporation). Similar to the fatigue testing, a metallic hemispherical cap was temporarily cemented (Provy; Dentsply Brazil) on each Y-TZP abutment, ensuring that the loading center was at a longitudinal distance of 11 mm. The single-load-to-fracture test was carried out at a crosshead speed of 0.5 mm/min until the fracture of the specimen. The load to failure (N) of each specimen was recorded. The failure mode was observed using stereomicroscopy (SteREO Discovery v20; Zeiss).

To illustrate the surface microstructure, representative abutments were examined on the flat surfaces at the lingual aspect with high-resolution scanning electron microscopy (SEM) (JEOL JSM-7500F; JEOL Ltd) field emission with an accelerating voltage of 2.00 kV. After aging, the abutments were cleaned in isopropyl alcohol for 10 minutes with an ultrasonic cleaner and stored under dry conditions at 37°C for 24 hours. Each abutment was coated with evaporated carbon and analyzed by secondary electron emission. The chemical composition of the as-received (as provided by the manufacturer) abutment was assessed with an energy dispersive spectrometer with an accelerating voltage of 8.00 kV.

A statistical analysis of the fracture load data was performed with software (IBM SPSS Statistics v20.0; IBM Corp). The approximate normality of data distribution was tested by the Shapiro-Wilk test. The 1-way analysis of variance (ANOVA) followed by the Tukey honest significant differences test were used to detect significant differences among the group means (α=.05).

Table 1. Phase content (wt%) of experimental groups

	MC Condition		AUT Condition		TC Condition	
Specimen	*t*-phase	*m*-phase	*t*-phase	*m*-phase	*t*-phase	*m*-phase
1	-	-	88.7 (5)	11.3 (5)	88.7 (6)	11.3 (6)
2	85.6 (4)	14.4 (4)	-	-	-	-
3	85.3 (5)	14.7 (5)	-	-	89.5 (5)	10.5 (5)
4	-	-	89.2 (5)	10.8 (5)	-	-
5	-	-	-	-	-	-
6	-	-	89.9 (6)	10.1 (6)	-	-
7	82.5 (5)	17.5 (5)	-	-	88.6 (5)	11.4 (5)
8	80.7 (5)	19.3 (5)	86.3 (5)	13.7 (5)	-	-

AUT, autoclaving; MC, mechanical cycling; TC, thermal cycling; *t-m* phases, tetragonal-to-monoclinic phase transformation.

RESULTS

XRD analysis revealed no detectable monoclinic phase in as-received Y-TZP abutments. Under aging conditions, monoclinic content was observed in some specimens of each group (Table 1). Figure 1 shows the Rietveld plot representing the observed data.

The mean fracture load values and standard deviations are shown in Table 2. ANOVA identified a significant group effect (P<.001). Table 3 summarizes the ANOVA. Results from the Tukey test showed that the control group had significantly higher resistance to fracture than the test groups (P<.001). Among the test groups, the highest decrease in fracture load was observed for the MC condition, whereas the lowest decrease was observed for the AUT condition, with significant differences between them (P=.034). The TC condition had no significant differences compared with the other test groups (P>.05). Stereomicroscopy analysis revealed that the predominant failure mode was abutment fracture at the connecting region (Fig. 2).

The chemical composition of the as-received abutment is presented in Figure 3. High-resolution SEM inspection of the as-received abutment revealed that

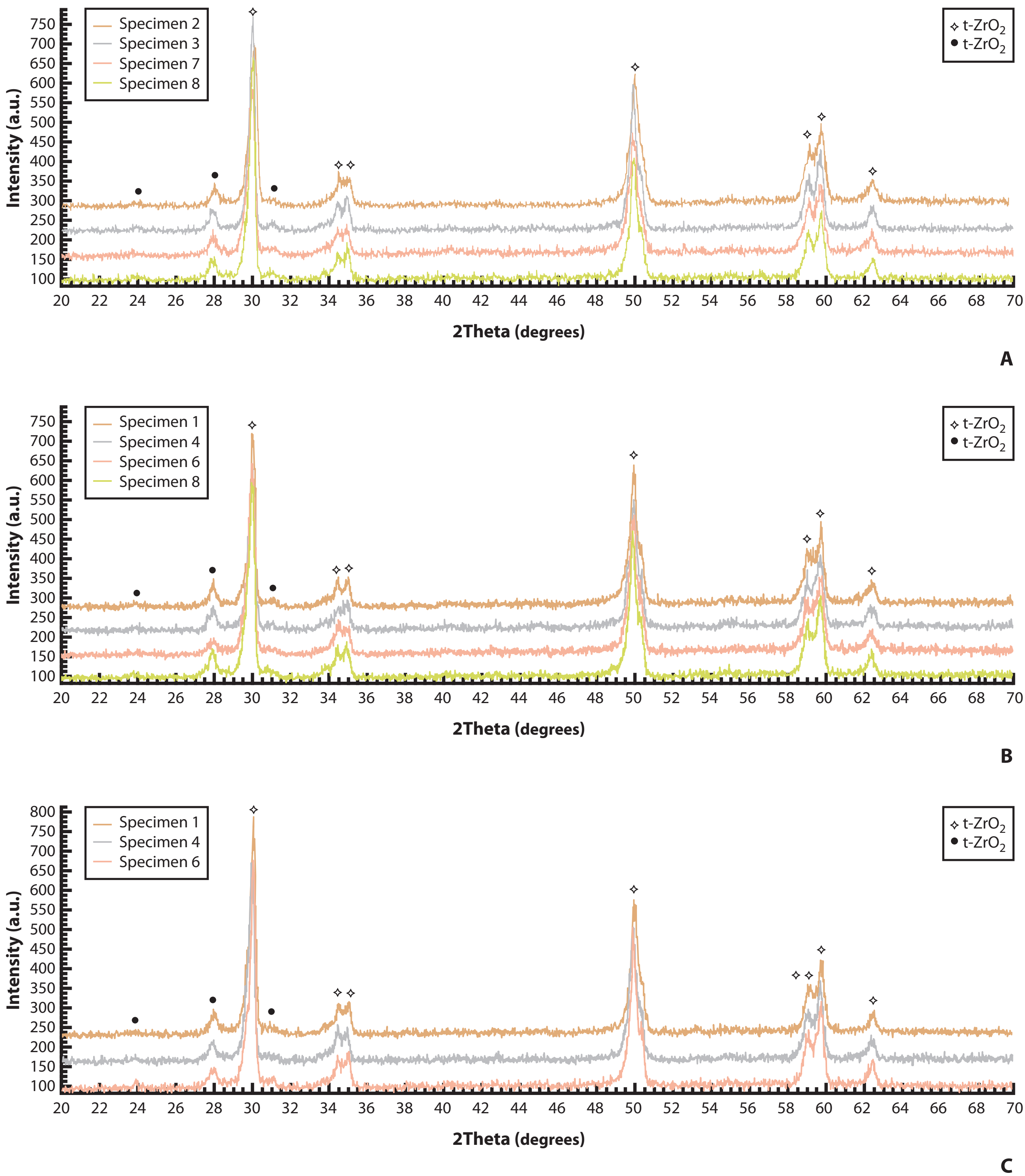

Figure 1. XRD diffraction patterns representing observed data. A, MC condition. B, AUT condition. AUT, autoclaving; MC, mechanical cycling; TC, thermal cycling; XRD, x-ray diffraction (analysis).

the surface milled by means of the computer-aided design and computer-aided manufacture (CAD-CAM) system was rough and covered with smeared materials and worn debris (Fig. 4A). Detailed inspection revealed plastically deformed zones at which the grain boundaries were not clearly visible (Fig. 4B). No differences were found in the process of surface degradation among the control and aged groups; however, all

Table 2. Means ±standard deviations of fracture load (N) measurements

Control	MC Condition	AUT Condition	TC Condition
721.77[a]	580.85[c]	636.78[b]	631.67[bc]
±58.62	±19.51	±29.73	±34.88

Same superscript letters indicate no statistically significant differences (P>.05).

Table 3. Results of 1-way ANOVA

Effect	*df*	F	*P*
Between groups	3	18.448	<.001*
Within groups	28		
Total	31		

*Statistically significant at P<.05.

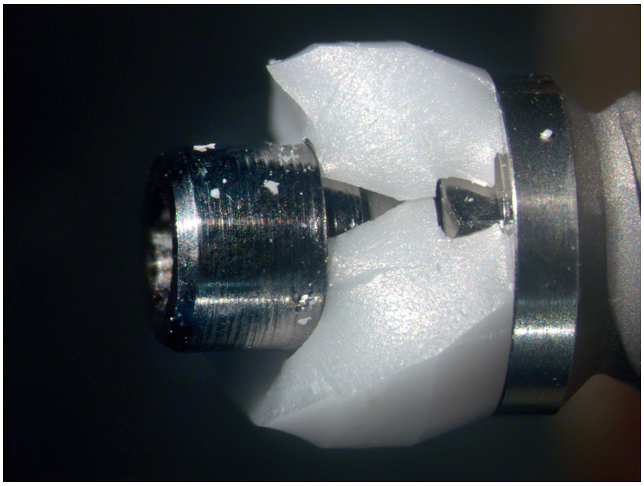

Figure 2. Image showing abutment fracture at connecting region.

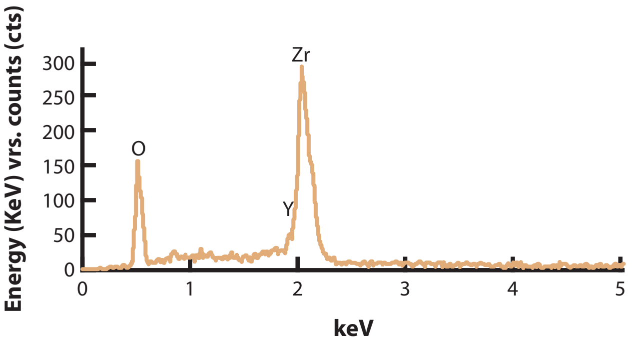

Figure 3. Chemical composition of as-received abutment determined by energy dispersive spectroscopy showing zirconia-based material matrix.

abutments presented grinding-induced surface flaws with internal microcracks penetrating the bulk of the material (Fig. 5). Moreover, worn grains with an irregular shape and even pieces of broken grains were randomly distributed on the surface.

DISCUSSION

Aging of Y-TZP is usually conducted in an autoclave, where the effect of the pressure of water vapor, the temperature, and the elapsed time represent the only basis for estimating the degradation rates of Y-TZP and, therefore, its lifetime. A risk assumption is a lifetime prediction according to the current ISO 13356 because the activation energy for *t-m* transformation in a given material cannot be obtained from such a "single data point" procedure. The lifetime prediction also cannot be considered to be approximately the same for all stabilized zirconia.[5,28] The present set results showed that all aging conditions induced the process of phase transformation (*t-m*) and led to a significant decrease in Y-TZP mechanical resistance compared with the control.

Interestingly, the MC condition, including the simultaneous effect of water and cyclic loading, degraded the fracture resistance significantly more than the AUT condition. Although hydrothermal condition can induce the *t-m* transformation with associated degradation of the mechanical properties,[23,24] Y-TZP, like all ceramic materials, is sensitive to subcritical crack growth. Natural cracks within the component's microstructure can propagate at a low rate when subjected to cyclic stresses in a humid environment.[16,17] As a result, an increase in the critical size defect could have accelerated the fracture process. Cotes et al[44] observed similar findings in Y-TZP disk-shaped specimens in which mechanical cycling for 15×10^6 cycles and thermo-mechanical cycling for 1.2×10^6 cycles decreased the flexural strength more than autoclaving (134°C/0.2 MPa/12 h). Similarly, as pointed out by Cotes et al,[44] the results of the present study also show that the decrease in Y-TZP mechanical resistance was not only the result of hydrothermal degradation. Therefore, mechanical aging might result in a more significant clinical representation than the steam autoclaving.

Although repeated thermal stresses may lead to a decrease in Y-TZP resistance,[39] the reduction of fracture load was also observed after TC condition. The thermal stressing may have led to tensions within the material and slow subcritical crack growth. Cotes et al[44] noted that thermal variations might contribute to fatigue and degradation only when associated with mechanical load. In another study with Y-TZP disk-shaped specimens, aging conditions such as thermal cycling (1×10^4, between 5°C and 55°C) and mechanical cycling (10^6 and 5×10^6 cycles) did not lead to significant changes in fracture strength.[45] These contradictions emphasize the assumption that aging sensitivity should be specifically determined for each Y-TZP ceramic, since aging is primarily dependent on microstructural characteristics.[21,22] Moreover, as stated in previous publications, different surface features lead to highly different aging sensitivity.[25-28] Thus, the Y-TZP resistance and fracture process of polished disk specimens may, to a large extent, be

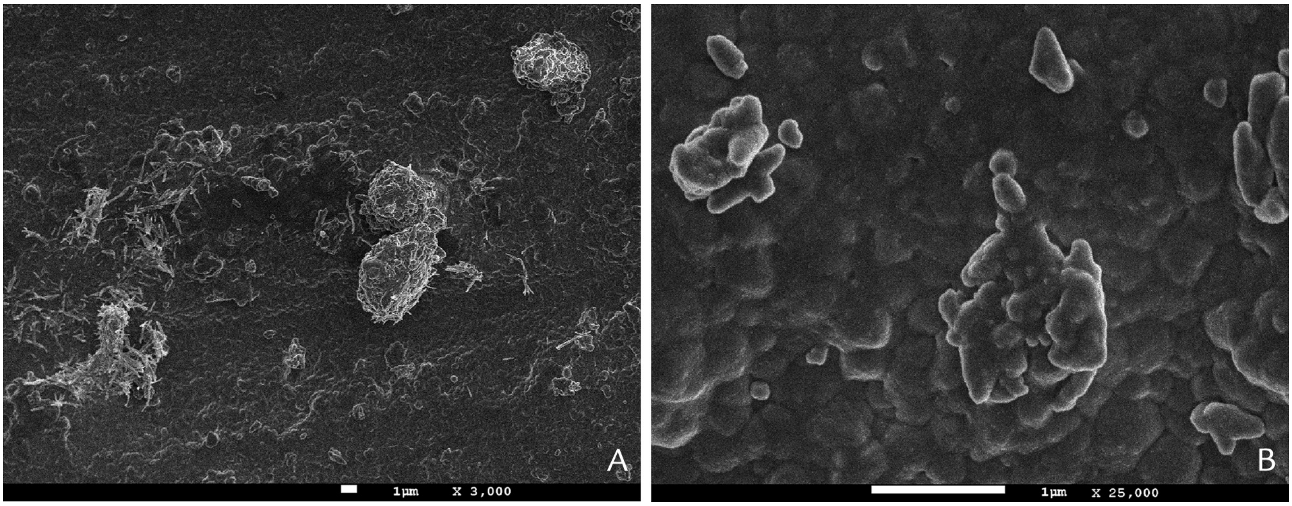

Figure 4. Scanning electron image of as-received abutment. A, Noticeable roughness and worn debris on surface (original magnification ×3000). B, Higher magnification shows representative features of plastic deformation (original magnification ×25 000).

different from that of abutments with a CAD-CAM-machined surface.

Monoclinic content was observed in all groups after aging; however, it occurred only in some abutments of each group. Thus, no correlation was found between the monoclinic phase content and fracture resistance. Kim et al[23] evaluated degradation of the flexural strength of Y-TZP ceramic after different low-temperature treatments and reported that monoclinic content increased sharply from 12% to 75% at the same time that flexural strength began to decrease. The present study recorded a remarkable reduction of fracture load values at low monoclinic percentages or even at the absence of the monoclinic phase. This may be attributed mainly to some limitations of the XRD analysis. Although well established and widely used to follow the transformation propagation quantitatively, the XRD method provides only monoclinic content over the penetration depth of the x-rays.[28] Furthermore, the x-ray beam is a few millimeters wide on most of the available instruments, and therefore, XRD extracts the monoclinic content relative to the Y-TZP local phase and not to the overall material.[5,46] Raman spectroscopy, as an example, seems to extract values of monoclinic content that are different from those extracted by XRD.[29] In this regard, further studies with advanced and accurate techniques are required to fully explain the correlation between the amount of monoclinic content and strength degradation.

Most studies focus on the damage on flat specimens and thus little is known about Y-TZP prosthetic restorations such as Y-TZP abutment-like complex geometries. CAD-CAM technology allows dentists to design and fabricate such components with a high level of accuracy and precision. However, it creates surface features such as roughness, plastic deformation, damage, and residual stress induced by the subtractive milling or grinding operations.[47] In this regard, fatigue is a specific problem as it can provide the energy to propagate an existing damage and also create new damage modes when combined with the wet environment.[16] Kim et al[26] demonstrated that CAD-CAM-prepared surface had pronounced effects on the hydrothermal degradation behavior of Y-TZP as a result of the CAD-CAM machining damage and the absence of surface compressive stresses in the fully sintered material. Thus, the rough surface, characterized by flaws and microcracks among the grains in this study, might be responsible for the reduction in Y-TZP mechanical resistance (Fig. 4). In addition to Y-TZP-based materials being susceptible to fatigue and hydrothermal degradation, tightening the retention screw may also induce high wedging forces inside the Y-TZP abutments.[14] Therefore, the exact mechanisms that control the strength reliability of such components have yet to be well defined.

In any load-to-fracture investigation, a clinical scenario must be considered. The lowest fracture resistance was 580.85 N for the MC condition, and this average was still higher than the maximal occlusal force in the anterior dentition, which ranged from 140 to 210 N.[48] However, clinical caution is indicated for posterior restorations since the highest fracture resistance was 721.77 N for the control group and the maximal occlusal force in this region may be as high 720 N.[49]

The failure mode analysis revealed a general pattern of crack extension at the connecting area (Fig. 2). Abutment fracture at the connecting area was also the main type of fracture found for Y-TZP abutments with external implant-abutment connection in many in vitro

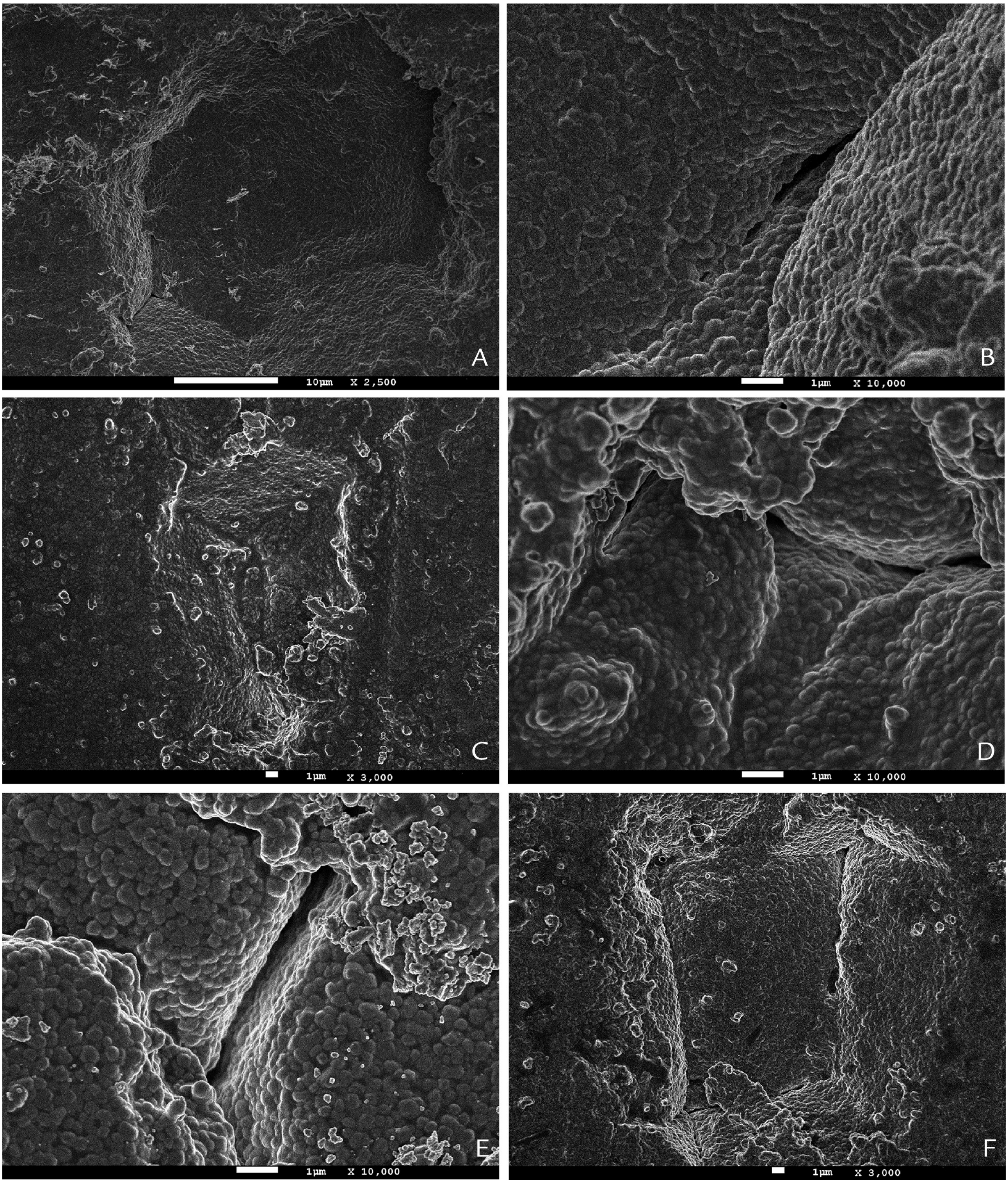

Figure 5. Scanning electron microscope image of as-received abutment and aged abutments showing deep surface flaw with internal microcracks. A, As-received abutment (original magnification ×2500). B, As-received abutment (original magnification ×10 000). C, MC condition (original magnification ×3000). D, MC Condition (original magnification ×10 000). E, AUT condition (original magnification ×10 000). F, TC condition (original magnification ×3000). AUT, autoclaving; MC, mechanical cycling; TC, thermal cycling.

studies.[10,36,50,51] Therefore, the abutment screw head may have affected the occurrence of fractures. This abutment type, available only with an external connection, was chosen because the XRD analysis requires a flat surface to observe and quantify the *t-m* phase transformation, which was found at the lingual aspect of the abutments.

CONCLUSIONS

Within the limitations of this study, the following can be concluded:

1. The resistance to fracture of Y-TZP implant abutments was susceptible to hydrothermal and mechanical conditions.
2. Hydrothermal and mechanical conditions induced phase transformation from tetragonal to monoclinical phase.
3. The surface microstructure of Y-TZP abutments did not change after being subjected to aging conditions.

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Noteworthy Abstracts of the Current Literature

Minimizing excess cement in implant-supported fixed restorations using an extraoral replica technique: A prospective 1-year study

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Purpose. Cementation of implant-supported restorations poses two major challenges: (1) minimizing excess cement (reducing the risk of peri-implantitis), and (2) establishing sufficient retention (reducing the risk of decementation). This study presents the first data on a clinical cementation technique that might address both problems.

Materials and Methods. Between 2011 and 2013, 39 patients were provided with 52 implants supporting 52 single crowns (SCs). All restorations were cemented extraorally using replicas made of pattern resin and zinc oxide cement. All decementation events and the peri-implant soft tissue status were assessed and compared with those from a group of 29 patients with 40 conventionally cemented SCs (control).

Results. In the experimental group, after 12 months, decementation was recorded in three individuals (7.69%) with 3 SCs (5.77%). In the control group, after 12 months, no case of decementation was recorded. No cases of peri-implantitis were detected in either group.

Conclusion. Within the limitations of this study, the authors conclude that the use of zinc oxide cement initially establishes sufficient retention of implant-supported fixed restorations independent of conventional or replica cementation techniques.