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RESEARCH AND EDUCATION

Zirconia changes after grinding and regeneration firing

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ABSTRACT

Statement of problem. Despite improvements in computer-aided design and computer-aided manufacturing (CAD-CAM) systems, grinding during either laboratory procedures or clinical adjustments is often needed to modify the shape of 3 mol(%) yttria-tetragonal zirconia polycrystal (3Y-TZP) restorations. However, the best way to achieve adjustment is unclear.

Purpose. The purpose of this in vitro study was to evaluate the microstructural and crystallographic phase changes, flexural strength, and Weibull modulus of a 3Y-TZP zirconia after grinding with or without water cooling and regeneration firing.

Material and methods. Ninety-six bar-shaped specimens were obtained and divided as follows: as-sintered, control; as-sintered with regeneration firing; grinding without water cooling; grinding and regeneration firing with water cooling; and grinding and regeneration firing. Grinding (0.3 mm) was performed with a 150-μm diamond rotary instrument in a high-speed handpiece. For regeneration firing, the specimens were annealed at 1000°C for 30 minutes. The crystalline phases were evaluated by using x-ray powder diffraction. A 4-point bending test was conducted (10 kN; 0.5 mm/min). The Weibull modulus was used to analyze strength reliability. The microstructure was analyzed by scanning electron microscopy. Data from the flexural strength test were evaluated using the Kruskal-Wallis and Dunn tests (α=.05).

Results. Tetragonal-to-monoclinic phase transformation was identified in the ground specimens; R regeneration firing groups showed only the tetragonal phase. The median flexural strength of as-sintered specimens was 642.0; 699.3 MPa for as-sintered specimens with regeneration firing; 770.1 MPa for grinding and water-cooled specimens; 727.3 MPa for specimens produced using water-cooled grinding and regeneration firing; 859.9 MPa for those produced by grinding; and 764.6 for those produced by grinding and regeneration firing; with statistically higher values for the ground groups. The regenerative firing did not affect the flexural strength. Weibull modulus values ranged from 5.3 to 12.4. The SEM images showed semicircular cracks after grinding.

Conclusions. Adjustments by grinding in 3Y-TZP frameworks should be performed with water cooling, and regeneration firing should be undertaken to obtain a more reliable material. (J Prosthet Dent 2017;118:61-68)

Zirconia has become a popular dental ceramic because of its suitable mechanical properties,[1,2] chemical stability, and esthetics.[3] Zirconia has 3 different crystallographic phases: monoclinic (*m*), at room temperature to 1170°C; tetragonal (*t*), at 1170°C to 2370°C; and cubic (*c*), at temperatures greater than 2370°C.[4-7] During cooling, at approximately 970°C, a *t*-to-*m* (*t-m*) transformation occurs, followed by volumetric expansion of 3% to 5%,[4,6,8] causing spontaneous fracture of the material. To avoid this transformation, oxides such as yttria, ceria, and magnesia can be added to the zirconia to stabilize the tetragonal phase at room temperature.[4]

However, under external mechanical stress, a *t-m* transformation can occur even in metastable zirconia when a microcrack is formed. Tetragonal crystals around the microcrack become *m* because of the stress induced by the growing microcrack, followed by volumetric expansion that generates compressive stress at the tip of the crack. This phenomenon, known as transformation toughening, demands more energy to propagate the cracks.[9]

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Clinical Implications

Adjustments to 3Y-TZP ceramics are often necessary clinically, but, depending on how they are performed, can decrease the mechanical properties and reliability of the material.

In dentistry, zirconia is used for crowns and metal-free partial fixed dental prostheses,[10-12] implants and abutments,[13-16] and orthodontic brackets.[17] Crowns and partial fixed dental prostheses are obtained by using a computer-aided design and computer-aided machining (CAD-CAM) technology, the precision of which has improved but which has not eliminated the need for some laboratory and intraoral adjustments.[18-24] Nakamura et al[25] reported that these adjustments could change the strength of the material, suggesting that more studies of this topic are needed.

Garvie et al[4] first reported the strengthening of zirconia-based ceramics after grinding because of the association between *t-m* transformation and the generation of superficial compressive stress. However, controversies persist regarding the influence of grinding and polishing on flexural strength because they have been correlated with the percentage of volume transformation, the severity of grinding,[26] the temperature locally generated by grinding, the metastability of the *t-m* transformation,[27-30] and whether cyclic conditions are involved.[31]

Considering that grinding can exert positive[32,33] or negative[34-36] effects on 3 mol(%) yttria-tetragonal zirconia polycrystal (3Y-TZP) ceramics, manufacturers have recommended different surface treatments,[37] including annealing,[32,38] which can act as a type of "regenerative firing" for the reestablishment of the tetragonal lattice,[39,40] thus increasing the reliability of the material. When heat treatments are used, the reverse transformation, *m-t*, can occur, and the compressive stress can be relieved, decreasing the flexural strength.[8,28] Despite this decrease in strength, the material may become more stable.[41]

The purpose of this study was to investigate the effects of grinding and regeneration firing on the microstructural and crystallographic phase changes, flexural strength (FS), and Weibull modulus of a 3Y-TZP ceramic. The null hypothesis was that the grinding and regeneration firing procedures would not affect the evaluated properties.

MATERIAL AND METHODS

Zirconia blocks (Lava Frame; 3M ESPE) were cut using a high-precision sectioning saw (IsoMet 1000; Buehler) with a diamond disk (Diamond Wafering Blade, series 15LC Diamond; Buehler) under water cooling to obtain 96 bar-shaped specimens of 2 dimensions: 25×5.0×1.5 mm and 25×5.0×1.9 mm. Edges were shaped by using a ceramic polisher (Exa Cerapol 0361HP; Labordental Ltda) to remove irregularities. Specimens were polished with silicon carbide paper (1400 and 2000 grit, 401Q; 3M) before sintering.

Table 1. Distribution of experimental groups

Group	Condition
AS	As-sintered, control
ASR	As-sintered with regenerative firing
DG	Dry grinding without regenerative firing
DGR	Dry grinding with regenerative firing
WG	Wet grinding without regenerative firing
WGR	Wet grinding with regenerative firing

The sintering procedure was performed according to the manufacturer's instructions at 1500°C for 8 hours in the Lava Furnace 200 (Dekema Dental-Keramiköfen GmbH). The specimens were divided into 6 groups (n=16) according to the treatment (Table 1). The final dimensions were 20×4.0×1.2 mm for the as-sintered (AS), control group and 20×4.0×1.5 mm for the ground (G) groups.

Grinding was performed using a coarse grit (150 μm) cylindrical diamond rotary instrument (4ZR; Komet) in a high-speed dental handpiece (KaVo Extra Torque 605; Kavo do Brazil Ind. e Com. Ltd) at 350 000 rpm, with (W) or without (D) water-cooling spray and mounted in a custom-made device (Fig. 1) to standardize the amount of longitudinal grinding (0.3 mm). The diamond rotary instrument was positioned parallel to the horizontal plane in contact with the specimens. The final dimensions (±0.01) were confirmed by using digital calipers (500-144B; Mitutoyo Sul Americana). After they were ground, the specimens were ultrasonically cleaned in isopropyl alcohol for 3 minutes and dried with paper towels.

The regeneration firing (R) was conducted using a porcelain furnace (AluminiPress; EDG Equipamentos e Controles Ltd) at 1000°C for 30 minutes with a 1000°C-predrying temperature and a heating rate of 1.36°C/min. After this procedure, the specimens were cooled to room temperature.

Thermal solid-state analysis, x-ray powder diffraction (XRPD), was used to identify the crystalline phases of the 3Y-TZP ceramic. Specimens were analyzed in an x-ray diffractometer (XRD-6000; Shimadzu Corp) at the 2θ range between 20 and 40 degrees with a step size of 0.05 degrees in continuous mode for 3 seconds. The peaks were identified by comparison with standard data files from the Joint Committee on Powder Diffraction Standards (JCPDS) International Centre for Diffraction Data.

A 4-point bending test was performed in artificial saliva at 37°C with a MTS model 810 testing machine (Material Testing System) equipped with a 10-kN load

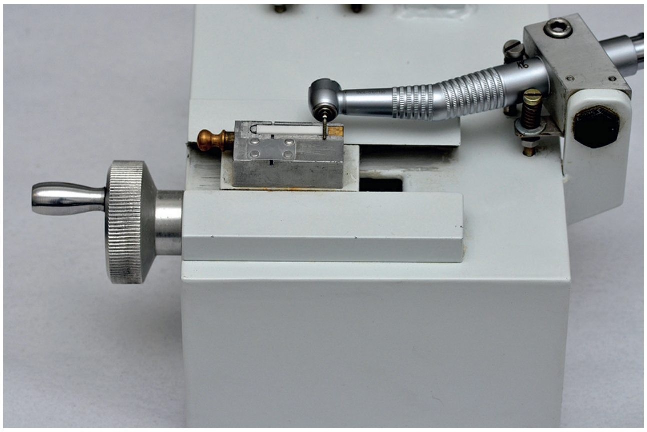

Figure 1. Grinding apparatus.

cell at 0.5 mm/min.[30,42] The FS values were calculated based on the formula [$\sigma=3PL/4wb^2$], where σ is the FS in MPa, P is the fracture strength in N, L is the distance between the external supports (span), w is the width of the specimens in millimeters, and b is the thickness of the specimens in millimeters.[42] The Kruskal-Wallis test and Dunn tests were used to analyze the data (α=.05).

The Weibull modulus (m) was used to evaluate the reliability of the strength among groups. The equation [$P(\sigma)=1-\exp(-\sigma/\sigma_0)^m$] allowed for calculation of the Weibull modulus, where $P(\sigma)$ is the fracture probability, σ is the fracture strength at a given $P(\sigma)$, σ_0 is the characteristic strength, and m is the Weibull modulus, which is the slope of the 1n (1n 1/1-P) versus in σ plots.[43,44]

The specimens were analyzed after cleaning with isopropyl alcohol, drying, and gold coating.[30] Then, microstructural surfaces were examined by using scanning electron microscopy (SEM) (JSM-6510LV; JEOL) at ×100 and ×150 magnification to detect morphological changes in longitudinal and lateral views.

RESULTS

Figure 1 shows the XRPD diffraction patterns of the experimental groups. The mean tetragonal peak (111) was identified in all specimens. Nonground specimens (Fig. 2A, B) had only the tetragonal phase. The ground surfaces showed a left shoulder that was identified as the monoclinical phase (Fig. 2C, E). The monoclinic phase was eliminated after the regeneration firing (Fig. 2D, F).

The results for flexural strength are shown in Table 2. Grinding increased the flexural strength of the specimens not subjected to regeneration firing. After regeneration firing (R), no significant differences were found among the experimental groups. Water cooling did not affect this property. However, when the Weibull modulus (Table 3) was analyzed, the reliability was seen to decrease substantially for groups without water cooling. For the G groups, the regeneration firing increased the strength reliability.

SEM images of each group are shown in Figure 3 (longitudinal view) and Figure 4 (lateral view). Specimens without grinding (Fig. 3A, B) showed uniform surfaces with minor scratches caused by the polishing procedure. After grinding, the scratches were intensified (Fig. 3C-F). In this view, regardless of the regenerative treatment and the use of water spray, no significant differences were found. In the lateral view, the G group exhibited semicircular cracks beyond the surface (Fig. 4).

DISCUSSION

The null hypothesis was rejected, as ground specimens showed higher FS than AS groups and wet grinding increased the material reliability, whereas dry grinding decreased it. Regeneration firing increased the ground material reliability and caused a monoclinic to tetragonal phase transformation.

Grinding is a common procedure in clinical practice and is used to achieve the best fit between sintered Y-TZP and the tooth preparation and to obtain enough space to apply the veneering porcelain.[15,18,19,34,38,44] This procedure can induce the *t-m* transformation, creating compressive stress[38] by the volume expansion of transformed crystals, which can increase the 3Y-TZP flexural strength.[9,41] In the present study, the monoclinical phase was detected (Fig. 2) in the ground specimens (grinding with water cooling [WG] and grinding without water cooling [DG] groups). The number of transformed crystals is proportional to the grinding severity,[20,41] and the volume and area of the transformed zone[8] are dependent on the method of specimen preparation.[44]

Grinding can create superficial flaws such as semicircular cracks, as shown in this study, in addition to grooves, microfissures, and fissures, depending on the diamond size, force, and rotation speed.[1,7,19,30,35,41,43,44] These cracks can propagate into the bulk of the material, decreasing its flexural strength.[29,32,44] Cracks may decrease the flexural strength of zirconia, when their depth is greater than the compressive stress layer of approximately 15 to 20 μm.[19,32] However, the ultimate flexural strength of the material depends on the balance between the increased flexural strength by toughening mechanism and the decrease in this property due to critical defects.

This investigation showed an increased flexural strength after grinding, regardless of water cooling, corroborating previous studies.[21,32,33,37,41] Conversely, other authors have reported decreases[8,28-30,34-36,44] or no changes[19,22] in this property after grinding. The highest values of flexural strength in ground specimens could be attributed to the *t-m* transformation (Fig. 2C, E) and to the development of compressive stress without deep cracks.

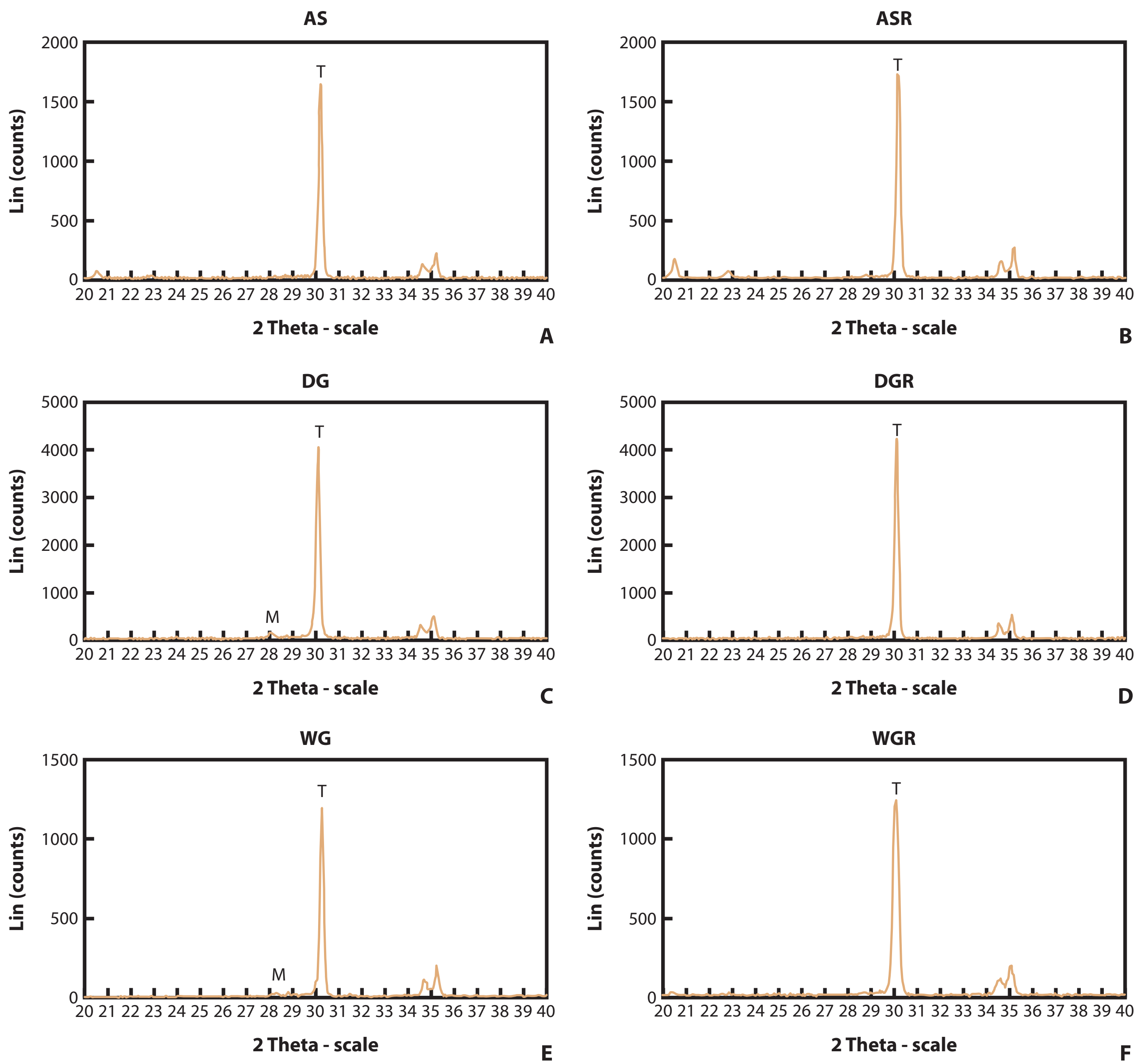

Figure 2. X-ray powder diffraction patterns of experimental groups: A, As-sintered control (AS). B, As-sintered with regenerative firing (ASR). C, Dry grinding without regenerative firing (DG). D, Dry grinding with regenerative firing (DGR). E, WG, wet grinding without regenerative firing (WG). F, Wet grinding with regenerative firing (WGR).

Table 2. Medians of flexural strength (MPa) and results of Kruskal-Wallis test

Condition	No Regenerative Firing	Regenerative Firing (R)	*P*
As-sintered (AS)	642.0 (577.4, 703.6)^{Aa}	699.3 (621.1, 766.9)^{Aa}	.070
Dry grinding (DG)	859.9 (704.9, 894.1)^{Ba}	764.6 (690.5, 868.5)^{Aa}	.624
Wet grinding (WG)	770.1 (721.2, 799.6)^{Ba}	727.3 (703.2, 758.2)^{Aa}	.122
P	.0004	.1441	

Values in parentheses first quartile and third quartile, respectively. Vertically, same superscript uppercase letters indicate no significant difference among groups ($P \geq .05$). Horizontally, same superscript lowercase letters indicate no significant differences ($P \geq .05$).

Table 3. Weibull modulus of experimental groups

Group	Weibull Modulus
AS	7.5
ASR	7.0
DG	5.3
DGR	6.0
WG	11.8
WGR	12.4

AS, as-sintered, control; ASR, as-sintered with regenerative firing; DG, dry grinding without regenerative firing; DGR, dry grinding with regenerative firing; WGR, wet grinding with regenerative firing.

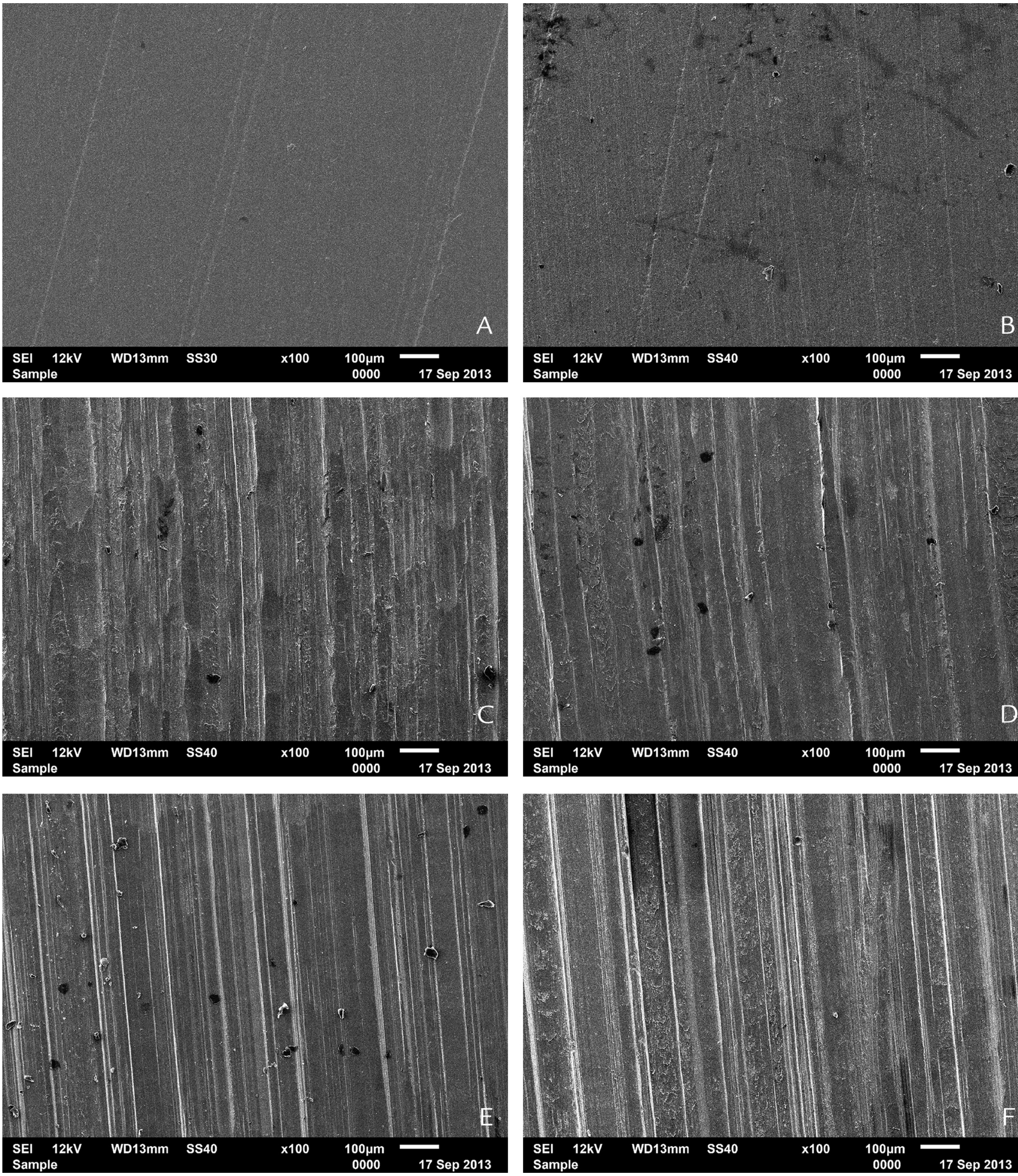

Figure 3. Scanning electron micrographs (original magnification ×100) of surfaces in longitudinal view. A, As-sintered. B, As-sintered with regenerative firing. C, Dry grinding without regenerative firing. D, Dry grinding with regenerative firing. E, WG Wet grinding without regenerative firing. F, Wet grinding with regenerative firing.

Regeneration firing, despite being able to reverse the phase transformation (*m-t*) (Fig. 2D, F) of the Y-TZP, did not significantly change its flexural strength, as observed by some authors.[32,41] Heat treatments might be able to relieve compressive stress, thus contributing to reduction of the flexural strength.[32,41] However, Ho et al[38] found that annealing of zirconia at 1100°C for 2 hours can decrease the extension of the cracks. In this study,

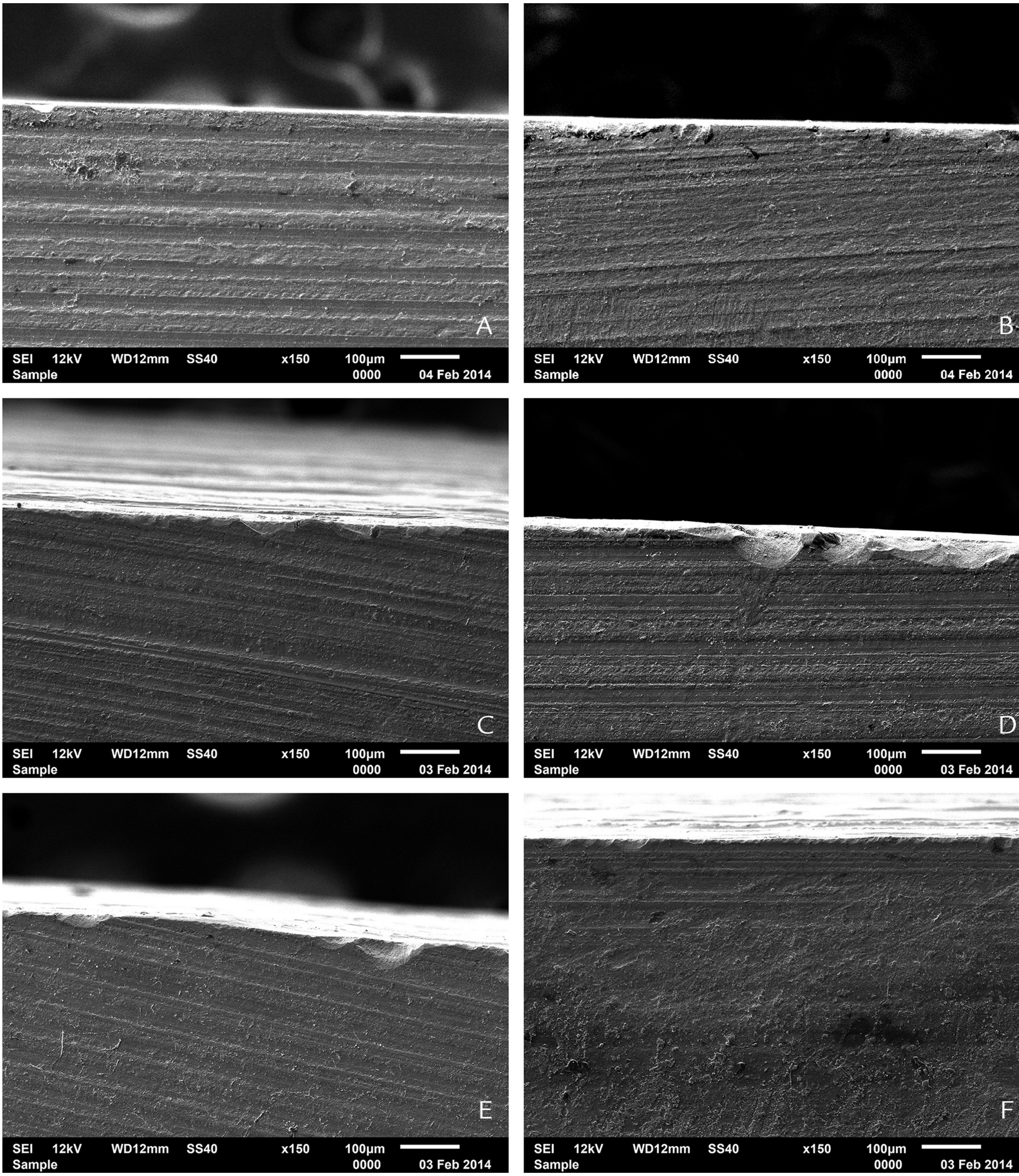

Figure 4. Scanning electron micrograph (original magnification ×150) of surfaces, perpendicular to ground. A, As-sintered. B, As-sintered with regenerative firing. C, Dry grinding without regenerative firing. D, Dry grinding with regenerative firing. E, Wet grinding without regenerative firing. F, Wet grinding with regenerative firing.

the maintenance of the flexural strength of the zirconia after regeneration firing may be explained by the interaction of reverse phase transformation and crack healing.

The grinding of fully sintered zirconia blocks produces prostheses with significant amount of the monoclinic phase.[2] This phase is associated with superficial micro-cracks, susceptibility to low-temperature degradation, and lower reliability.[22] These studies[2,22] showed the importance of controlling the final surface of Y-TZP for biomedical applications. In summary, although high strength might seem to be a good property for dentistry, long-term performance and reliability must be considered.[40] Strength data of ceramic materials usually show asymmetric distribution because of the materials' wide variability because of flaws incorporated during processing.[44] Thus, considering that grinding-induced flaws in the material, the Weibull modulus (Table 3), is as important as the flexural strength results found in this study.

According to some authors,[21,29] a low Weibull modulus is indicative of variations in the surface state, suggesting an increase in microcrack density within the damaged layer. This observation could explain the results for the DG (m=5.3) and DGR (m=6.0) groups. A low Weibull m value indicates an increase in flaw size population, decreasing the reliability of the materials and resulting in unexpected failures.[44] Except for the control group, regeneration firing increased the Weibull modulus, perhaps because it eliminated the compression layer that was induced by grinding.

The higher Weibull modulus (>10) found in the WG and WGR groups showed that grinding should be performed under irrigation when zirconia frameworks adjustments are necessary. Regeneration firing after grinding might be suggested, even that the flexural strength may decrease, as shown by some authors.[8,28-30,34-36,44] Based on the Weibull results, generally, the reliability of ground zirconia was increased after regeneration firing, and thus the achievement of a more reliable material may contribute to its clinical longevity. Despite the fact that grinding generally increases the results of monotonic strength tests of zirconia, their fatigue strength must be evaluated once under cyclic conditions, similar to those observed clinically, the crack formed during grinding can grow into the bulk of the material leading to a decrease in its mechanical properties.[31]

A limitation of this study was not evaluating long-term stability under cyclic loading conditions. Further studies should evaluate mechanically and thermally ground zirconia to ensure its longevity.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Adjustments by grinding Y-TZP frameworks should be performed with cooling under water.
2. Regeneration firing should be undertaken before the application of a porcelain veneer to achieve a more reliable material.

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Noteworthy Abstracts of the Current Literature

Toward optimizing dental implant performance: Surface characterization of Ti and TiZr implant materials

Murphy M, Walczak MS, Thomas AG, Silikas N, Berner S, Lindsay R

Dent Mater 2017;33:43-53

Objective. Targeting understanding enhanced osseointegration kinetics, the goal of this study was to characterize the surface morphology and composition of Ti and TiZr dental implant substrates subjected to one of two surface treatments developed by Straumann. These two treatments are typically known as SLA and SLActive, with the latter resulting in more rapid osseointegration.

Methods. A range of techniques was applied to characterize four different substrate/surface treatment combinations (TiSLA, TiSLActive, TiZrSLA, and TiZrSLActive). Contact angle measurements established their hydrophilic/hydrophobic nature. Surface morphology was probed with scanning electron microscopy. X-ray diffraction, Raman μ-spectroscopy, and X-ray photoelectron spectroscopy were used to elucidate the composition of the near-surface region.

Results. Consistent with previous work, surface morphology was found to differ only at the nanoscale, with both SLActive substrates displaying nano-protrusions. Spectroscopic data indicate that all substrates exhibit surface films of titanium oxide displaying near TiO2 stoichiometry. Raman μ-spectroscopy reveals that amorphous TiO2 is most likely the only phase present on TiSLA, whilst rutile-TiO2 is also evidenced on TiSLActive, TiZrSLA, and TiZrSLActive. For TiZr alloy substrates, there is no evidence of discrete phases of oxidized Zr. X-ray photoelectron spectra demonstrate that all samples are terminated by adventitious carbon, with it being somewhat thicker (∼1nm) on TiSLA and TiZrSLA.

Significance. Given previous in vivo studies, acquired data suggest that both nanoscale protrusions, and a thinner layer of adventitious carbon contribute to the more rapid osseointegration of SLActive dental implants. Composition of the surface oxide layer is apparently less important in determining osseointegration kinetics.