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Effect of finishing/polishing techniques and low temperature degradation on the surface topography, phase transformation and flexural strength of ultra-translucent ZrO_2 ceramic

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Abstract

Objective: To investigate the effect of different surface finishing and polishing regimes and low temperature degradation on flexural strength, phase transformation and surface topography of ultra-translucent ZrO_2 ceramic.

Methods: 300 (n=15/group) of conventional zirconia (Z: Ice Zirkon Transluzent) and ultra-translucent zirconia (UT: Prettau Anterior) bar-specimens were made and divided according to the

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

"Finishing/Polishing" - (C - Control, B - diamond rubber polishers, P - adjusting with burs, PB - adjusting with burs + diamond polishers, PG - adjusting with burs + glaze), "Low temperature Degradation (LTD)" (with or without a treatment at 127 °C, 1.7 bar/24h). Then, a 3-point mini flexural test was performed in a universal testing machine (1 mm/min, 500 kgf load cell). SEM, EDS, XDR, AFM, optical profilometry and Weibull analysis were performed. Data were analyzed by 3-way ANOVA and Tukey's post-test (5%).

Results: Groups ZPB_D (1670 ± 253 MPa), ZB_D (1664 ± 217 MPa), and ZB (1655 ± 3678 MPa) showed significantly higher flexural strength than the UTPG group (372 ± 56 MPa). The Weibull modulus was significantly higher for the ZP_D group compared to the UB, UC_D, UP_D and UPB_D, while UTB, UTC_D and UTP_D had the lowest value. Monoclinic phases were observed only in the conventional zirconia groups and were more evident after LTD. Diamond rubber polishers presented less roughness for both zirconias.

Significance: The use of diamond rubber polishers is the most suitable finishing/polishing method for zirconia ceramic restorations and that final glazing reduces the fracture resistance of these materials.

Keywords

Dental ceramics; Monolithic zirconia; Cubic zirconia; Monoclinic zirconia

1. INTRODUCTION

Yttria partially stabilized zirconia (Y-PSZ) is one of the most versatile ceramic materials used in dentistry because of their excellent mechanical and physical properties [1]. In recent years, this material has undergone microstructure and composition modifications to improve translucency without losing mechanical properties [2,3]. The first generation monolithic zirconia (3Y-TZP, 3 mol% yttria-stabilized zirconia polycrystals) had high opacity due to the amount and size of the Al_2O_3 (aluminum oxide) particles. In addition, the material had low structural stability and fracture resistance at high temperatures, and was thus discontinued for dental rehabilitation [4]. In 2012, a second generation of zirconia with reduced grain size and the amount of aluminum oxide sintering aids and higher translucency was introduced [5]. But its translucency was still incomparable to glass ceramics used for esthetic restorations.

Further structural modifications, such as reduction in aluminum oxide, increase of yttrium oxide (4 to 5%), and a cubic-to-tetragonal weight ratio of up to 25–75 [6,7] resulted in the third generation of zirconia, with high translucency and lower fracture resistance compared to previous generations, but better mechanical properties than glass ceramics. These characteristics allowed the ultra-translucent zirconia to be widely used in front teeth restorations such as crowns, veneers, and dental "laminate veneers" [8] made by CAD/CAM (computer-aided design/ computer-aided manufacturing).

Despite the accuracy of monolithic zirconia restorations made by CAD/CAM technology, small adjustments might often be necessary prior to the cementation for adequate interproximal and occlusal contacts, and overall contour improvement [9,10]. Due to the

hardness of zirconia, adjustments are usually made with diamond burs, which affect the glaze layer and original smoothness of the surface [11]. A rough surface also may cause greater wear of the antagonist tooth, favors biofilm accumulation and affects the surface characteristics make it irregular [12–14].

These changes lead to greater stress on the ceramic surface and indirectly increase the degradation of the material and decrease its esthetic and longevity [15,16,17]. In order to reestablish the smoothness and shine of monolithic zirconia surfaces, several techniques have been proposed, such as glazing [18], polishing with diamond rubber polishers [19] and the use of fine diamond burs [9]. Despite the benefits of a polished surface, some finishing and polishing methods can lead to the development of cracks that can propagate under traction forces [20], weakening the structure.

Among the methods described, application of glaze after adjustment has been questioned. Numerous studies have demonstrated a reduction in the fracture toughness of these ceramics [21,22] after their application and also an increase of the roughness. On the other hand, diamond rubber polishers have been one of the most used methods that produce restorations with better surface morphological characteristics as well as resistance to fracture [19].

The heating of the zirconia surface during adjustments can promote tetragonal-to-monoclinic phase transformation and affect the resistance to fracture of the material. However, this mechanism is still unclear [23]. In addition, the fracture resistance of the material can decrease when subjected to hydrothermal degradation and where the phase transformation zone has spread deep into zirconia. There is a formation of small surface defects that tend to propagate throughout the zirconia, affecting the mechanical properties. [23–25].

Currently, there is no established finishing and polishing protocol for monolithic ultra-translucent zirconia, and the effects on strength and surface characteristics of the available regimes are unknown. Thus, the objective of this study was to evaluate the effect of different finishing and polishing regimes and low temperature degradation on flexural strength, surface topography, and phase transformation of ultra-translucent zirconia. The hypotheses are: h1-hydrothermal degradation can reduce the flexural strength of zirconia, and h2- the adjustments can reduce the flexural strength of the ceramics.

2. MATERIALS AND METHODS

The materials (the trade name, manufacturers, chemical composition and batch number) used in this study are presented in Table 1.

2.1. Preparation of samples

The flowchart of the study design is shown in Figure 1. Disks (95 mm diameter and 18 mm height) of conventional zirconia (Z) (Ice Zirkon Translucent, Zirkonzahn, Gais, Italy) and ultra-translucent zirconia (UT) (Prettau Anterior, Zirkonzahn, Gais, Italy) were sectioned with double-sided diamond discs (22 × 0.15 mm, Dhpro, Paraná, Brazil) to obtain 300 zirconia bars of 10 × 2.5 mm, with 30 bars having a 1.3 mm height for the control group,

and 30 bars having from 1.7 to 2.1 mm for each experimental group. The bars were regularized with 800, 1000, and 1200-grit sand papers.

Prior to sintering, the samples were cleaned in an ultrasonic bath in distilled water for 5 minutes. With the 25% contraction from sintering (Zirkonofen 600 oven, Zirkonzahn, Gais, Italy) [26], the final dimensions of the bars were 8 × 2 × 1 mm (+/− 0.2 mm) for the control groups and 8 × 2 × 1.7–1.3 mm for the treatment groups [27]. After treatments, all the specimens had the same thickness of 1 mm. The samples were then divided into 20 groups according to the "Ceramics" factor (2 levels), "Finishing and polishing" factor (5 levels), and "Low temperature Degradation - LTD" factor (2 levels).

2.1.2. Finishing and polishing—The samples were adapted to a silicone matrix (Elite, Zhermack, Badia Polinese, Italy) for stabilization and divided into 5 groups:

C: no treatment;

P: smoothing with cylindrical ultra-fine diamond bur (# 4135-FG, 90–120μm, KG Sorensen, Cotia, Brazil) using a high-speed dental micro motor with water cooling and standard movements, until reaching a thickness of 1 mm. Burs were replaced after 10 samples.

B: polishing with abrasive rubber polishers of extra-hard (100 Shore A) diamond-impregnated polyurethane (Premium Compact, Dhpro, Paraná, Brazil). The disks were used according to the manufacturer's instruction (HZ1DL for wear, HZ2DL for pre-polishing, and HC3DL for high gloss) at 12,000 rpm (20 seconds per disk) and standard movements, until the surface was smooth and shiny.

PB: combination of burs and rubber polishers as described above.

PG: After burs, application of a single layer of glaze (Ivocolor fluor, Ivoclar, Schaan, Liechtenstein) using a brush followed by sintering.

2.1.3. Low-temperature degradation (LTD)—After the different finishing and polishing regimes, half of the samples of both zirconia groups ($n = 150$) were subjected to autoclave hydrothermal degradation (Cristófoli, Paraná, Brazil) for 24 h at 127°C and 1.7 bars [27].

2.2. Mini flexure resistance test

The zirconia samples were subjected to the three-point mini flexure resistance test in a universal testing machine (INSTRON, Norwood, Massachusetts, USA). An adjustable metal bending test device was used. The sample was supported by two rolls 6 mm apart with the treated side facing downwards [28]. Load was applied in the center with a loading rate of 1.0 mm/min and a load cell of 500 kgf [27]. The flexural strength in MPa was calculated at the time of failure, according to the equation:

$$RF = 31F/\left(2WH^2\right)$$

where l is the distance (mm) between the support rolls, F is the load (N) applied at the moment of failure, H is the height (mm) of the specimen, and W is the width of the specimen.

2.3. Atomic Force Microscopy

Forty blocks ($n = 2$) of each type of zirconia (4×4×1 mm) were made to perform the complementary analyzes. For the analysis of surface topography, the silicon tip of the probe was coated with gold (40 μm, 0.01 to 0.025 Ω.cm) and used in intermittent contact mode to obtain the 3D images. The scans were performed in an area of 5 × 5 μm (PPP-NCL probes nanosensors) with constant force of 48 N/m, and the images were processed with the Gwyddion™ software (v 2.33, GNU, Free Software Foundation, Boston, MA, USA).

2.4. X-rays diffraction

X-ray diffraction analysis was performed to evaluate the presence of the monoclinic (M) and tetragonal (T) phases (Equation A), determine the percentage of zirconia $t \rightarrow m$ transformations (Equation B) and the depth of the transformation zone (Equation C). The samples used in the atomic force microscopy (N = 40; $n = 2$) were analyzed in the diffractometer (D2Phaser, Bruker) using copper radiation (CuKα, λ = 1,54Å). The scans were performed with 10 mA current, 30kV, using a Lynxeye detector, 0.02 degree/step, and acquisition time of 0.1 s. The graphs were generated with Origin 8. Subsequently, the phase percentages were determined, where $(-111)_M$, 2θ= 28º; $(111)_M$, 2θ= 31.2^0; $(101)_T$. The equations used were:

$$X_M = (-111)_M + (111)_M / (-111)_M + (111)_M + (101)_T \quad \text{Equation A:}$$

$$f_M = 1.311 \times X_M / 1 + 0.311 \times X_M \quad \text{Equation B:}$$

$$PTZ = (sen\Theta / 2\mu) \times [In(1/1 - f_M)] \quad \text{Equation C:}$$

2.5. Optical profilometry

Eight samples of each group used for the flexural test (8×2×1 mm) had their surfaces evaluated in a 3D optical profilometer (Taylor Hobson-AMETEK, Leicester, England). An area of 300 × 300 μm was scanned and the mean of three readings was used for the roughness value (Ra) of each group (μm). 2D and 3D images of the surfaces were obtained.

2.6. Scanning Electron Microscopy / X-ray Dispersive Energy Spectrometry

Samples were gold-sputtered (BAL-TEC SCD 005) for 130 seconds at 15mA to obtain a 80 Å layer. An area of 6 μm with 5000x magnification was evaluated and the percentages of the main chemical elements were obtained. In addition, micrographs were obtained with the scanning electron microscope (SEM) (Hitachi, Tokyo, Japan) to verify the surface modifications promoted by the different treatments.

2.7. Statistical analysis

The power of the study was calculated using the OpenEpi website, considering a 95% confidence interval. Statistical assumptions were evaluated before statistical analysis. The results indicated that the residuals were normally distributed and, by plotting against predicted values, the uniformity was checked, then none of the ANOVA assumptions were violated (figure 2). Levene's test was performed and there was no statistically significant difference amongst the standard deviations (p = 0.647). These results relate that the data follow a normal distribution. Three-way analysis of variance (ANOVA) and Tukey's test (5%) were performed to compare flexural strength and roughness between groups. The computer program STATISTIX (Analytical Software Inc., version 8.0, 2003) was used for analyses.

Weibull analysis was performed to evaluate the reliability of the flexural strength test, using the Weibull parameter (m) and the characteristic strength (σ_o), with a confidence interval of 95%, being determined in a lnσc-ln [ln 1 / (1- $F(\sigma_c)$] diagram (according to ENV 843–5):

$$\mathrm{Lnln}(1/1 - \mathrm{F}(\sigma_c)) = m\ln\sigma_c - m\ln\sigma_o$$

The characteristic strength is the strength at a probability of approximately 63.3%, and the Weibull modulus is used as a measure of strength distribution, which expresses the structural homogeneity of the material. Statistical analysis was performed using the Minitab software (version 17, 2013, Minitab, State College, PA). The level of significance was 5%.

3. RESULTS

3.1. Mini flexural strength

The factors "LTD" (p = 0.01), "Finishing / Polishing" (p < 0.001), and "Zirconia" (p < 0.001) independently influenced the values of flexural strength. The interaction of "Polishing × Zirconia" (p = 0.0000) and "Polishing × Zirconia × LTD" (p = 0.02) were also statistically significant. (Table 2).

Conventional zirconia (1398 MPa) had higher flexural strength than ultra-translucent zirconia (528 MPa). LTD significantly increased the resistance of zirconia (1007 MPa) compared to samples not subjected to LTD (919MPa). Regarding the "Finishing / Polishing" factor, the use of rubber polishers promoted a higher flexural strength (1183.4MPa) than the groups treated with Bur + rubber Polisher (1066.4MPa), Control (1012MPa), Bur (933MPa), and Bur + Glaze (621MPa) - Tukey test.

The comparison between all the experimental groups (Table 3) showed that the ZPB_D (1670 ± 253), ZB_D (1664 ± 217) and ZB (1655 ± 3678) groups had the highest flexural strength, and were different from the other groups except ZP_D (1499 ± 134), ZPB (1497), ZC_D (1464 ± 258), and ZC (1456 ± 222). The ultra-translucent zirconia (UT) groups presented lower flexural strength. The UTB (624 ± 186), UTB_D (792± 169CD), UTC_D (679 ± 225) and $UTPB_D$ (602 ± 164) groups had significantly higher flexural strength values compared to the other groups, and UPG (372 ± 56) had the lowest value, which was significantly different

from the other groups, especially when compared to the groups treated with rubber polishers.

3.2. Weibull Analysis

The results of Weibull analysis are shown in Table 4 and Figure 3. The Weibull modulus (*m)* was significantly higher for the ZP_D group compared to the UB, UC_D, UP_D and UPB_D groups, which had the lowest value.

3.3. Atomic Force Microscopy

The control group of the UT zirconia not subjected to LTD (Figure 4) presented surfaces and grains that were more regular than the group submitted to LTD. In the groups treated with Burs + Polishing (UTPB) and Polishing (UTB), markings were observed, indicating the use of rubber polishers, and that surfaces lose uniformity after LTD.

3.4. X-Ray Diffraction (XDR)

Figure 5 shows the X-ray diffraction analysis for each group. For both types of zirconia, peaks for the tetragonal phase were detected. However, the monoclinic phase peaks were observed at the 28 ° and 31.2 ° angles only in the conventional zirconia groups, and were more evident after LTD. Only in the ZPG group a reduction of the monoclinic phase was observed after LTD.

The FM calculated after the X-ray diffraction analysis (Figure 6) showed that the percentage of monoclinic phase increased after LTD. However, in the ultra-translucent zirconia groups, no significant change occurred after LTD. In the ZPG group, a reverse phase transformation occurred after LTD with reduction of the monoclinic phase. Likewise, LTD promoted a greater depth of $T \rightarrow M$ phase transformation, except in the ZPG_D and UT zirconia groups (Figure 7).

3.5. Optical profilometry - roughness

The “finishing and polishing” factor independently influenced the roughness of the zirconia surface (Table 5). The polished groups (0.0356) and bur + polished groups (0.0508) presented lower roughness, differing statistically from the other groups. The bur-finished samples had the highest roughness values (0.5491) and were statistically different than the other groups (Table 6).

3.6. X-ray Dispersive Energy Spectrometry

The EDS analysis revealed that the control group presented the following elements: Zirconia (Zr), Aluminum (Al), Carbon (C), and Yttrium (Y). The weight percent for each chemical element found for ultra-translucent zirconia was 16.65% of C, 79.31% of Z, 0.24% for Al, and 3.79% for Y. For conventional zirconia the percentages were 13.55% of C, 86.25% of Z, and 0.20% of Al; Y was not detected due to its low molecular weight. Carbon was also detected in the finished and polished groups for both types of zirconia, as the diamond disks used for sample preparation and the finishing and polishing materials (polishers, diamond burs, and glaze) all have Carbon in their composition.

3.7. Optical profilometry - surface morphology

Figure 8 (A–J) shows the images obtained with optical profilometry for each of the finishing and polishing regimes. Peaks (reddish) and valleys (bluish) indicate the roughness that the finishing and polishing techniques promoted. The groups treated with bur-polishing (Figures 8E and F) show more pronounced peaks and valleys in relation to the other groups. The rubber-polished groups (Figures 8C and D) had surfaces that were more uniform. Glaze isles were observed on the surface of samples finished with burs followed by glaze application (Figures 8I and J), demonstrating that the applied glaze layer was not sufficient to cover the entire surface.

3.8. Scanning Electron Microscopy

The UTP group had a greater degradation of the surface compared to the conventional zirconia. A larger quantity of craters was present and regions where the material was detached from the surface were noticed, indicating that this procedure causes more damage to the surface than the others. In the control group, larger zirconia grains were seen in the UT compared to the conventional zirconia, and the LTD (UTC_D) groups presented a more irregular surface than their respective control (UTC). However, surface roughness was not significantly influenced by LTD, as previously noted. The SEM micrographs are show in the Figure 9.

4. DISCUSSION

Flexural strength is a very relevant property of friable materials such as ceramics, which are more fragile when subjected to tensile stresses than to compression [29]. Several fracture resistance tests have been used and recommended by the ISO - international organization for standardization 6872/2008 [30] to evaluate ceramic materials, such as three-point [31] (the most used), four-point [32], and biaxial flexural tests [33]. Because the specimens in this study were small, the mini-flexural test was used. The methodology was based on a previous study [27] that obtained similar values with the regular three-point flexural test and the mini-flexural test [34,35]. The advantages of using the mini-flexural test are the reduced size of samples and substrates, and the easiness with sample handling and individualization.

It is important to note that the current study adopted short beam geometries (8 × 2 × 1–2 mm) to determine the flexural strength due to restrictions in specimen availability. Therefore, some of the ISO 6872 specifications could not be met. According to a recent publication [36], friction between zirconia beam and supporting rollers can significantly reduce the measured flexural strength value relative to the prediction by beam theory. Such a reduction effect becomes more pronounced for beams with shorter dimensions. However, the actual frictional force is difficult to estimate in the present study since the specimen surface finish (which is a variable in this study) determines the friction coefficient. In light of the above analysis, we acknowledge that the present flexural strength data of zirconia with various surface treatments shall not be used for direct comparison with results reported in other studies.

This study evaluated the flexural strength of a conventional first-generation zirconia (Ice Zirkon transluzent) and an UT third-generation zirconia (Prettau anterior) used for CAD/CAM monolithic restorations. The flexural strength of the conventional zirconia was 1398 MPa, significantly higher than that of the UT (528 MPa). The results can be explained by changes in zirconia microstructure and composition to improve translucency that can influence the material mechanical resistance [3,37,6]. In the EDS chemical analysis, a greater amount of yttrium oxide was found in the UT zirconia compared to the conventional one. The cubic phase of the UT zirconia presents a crystalline arrangement of isotropic behavior. In such structure, light passes from one grain to another without scattering, increasing the zirconia translucency.

In this study, the first hypothesis proposed was rejected. The LTD also significantly increased the fracture resistance of zirconia. Although conflicting with some studies [25, 38, 39], there is evidence that hydrothermal degradation may increase the fracture resistance [28,40,41]. The degradation, simulated by autoclave moist heating, occurs in the zirconia crystals due to the spontaneous martensitic transformation from the tetragonal to the monoclinic phase when subjected to low heat in the presence of moisture. The volume increase of the monoclinic grains generates a protective layer of compressive stress, preventing the propagation of cracks and increasing the fracture resistance of the material. In addition, the phase transformation occurred on the surface of the zirconia, that is, there was no transformation in the deeper layers of the material. Thus, there was no reduction of the mechanical properties. This aspect can be observed in the depth of the phase transformation zone. Several other factors may also influence the resistance of zirconia [40], including grain size [17], sintering conditions [40, 42, 43], and different hydrothermal degradation protocols [44], which can increase phase transformations, as occurred in most of the groups, especially in conventional zirconia groups.

Phase transformations were also verified in X-ray diffraction analysis, in agreement with several studies [6,44–46]. In ultra-translucent zirconia, phase transformations were not observed, which is due to the composition of the material. The ultra-translucent zirconia is metastable in the tetragonal phase, contains to around 75% cubic phase [6], being considered a fully stable cubic / tetragonal zirconia. Stable levels of cubic phase are due to the higher amount of yttrium oxide (approximately by 8% mol). Therefore, transformations from tetragonal to monoclinic phase occur rarely due to the low availability of the tetragonal phase [47]. In some groups, LTD promoted an increase in grain size, which might be related to $T \rightarrow M$ phase transformation [48].

The “Finishing and Polishing” factor influenced the zirconia fracture resistance. The groups that presented the highest resistance were those treated with rubber polishers alone. The diamond impregnated polyurethane rubber polishers promoted a more uniform zirconia surface due to monoclinic phase transformation, generating a residual stress layer on the surface and thus increasing fracture resistance [49,50]. In addition, the slight warming generated by the polishing may also have induced phase transformation due to the heat generated by the rubbers [49].

The first polishing rubber disk of the polishing kit indicated by the manufacturer has high abrasiveness and can be used in place of the diamond bur for small adjustments as it removes a significant amount of material. The other disks are used for finishing and polishing the zirconia surface. In addition, SEM micrographs and profilometry images showed that polishing was the technique that provided the highest smoothness and lowest roughness (0.0356 ± 0.03) compared to the other techniques. It can be inferred that surfaces that are better polished present greater fracture resistance [51]. Hence, the second hypothesis proposed in this study was partially rejected.

On the other hand, the use of diamond burs for removing large amounts of ceramic material caused a reduction in flexural strength of zirconia, similarly to other studies [52–54]. This procedure can cause structural defects in areas of greater tension in the zirconia and create microcracks and fissures that can propagate into the material, leading to a degradation of the mechanical properties [55,56]. These characteristics were observed in the SEM images, which showed detachments of grains and chips, as well as in 3D profilometer images, which revealed the presence of high peaks and deep valleys. Several studies have also demonstrated through fractographic analysis that fractures in zirconia ceramic restorations originate from superficial/subsurface regions [57,58,59] and reported that various clinical/laboratory procedures on the occlusal surface of restorations may induce crack initiation and propagation that affect the mechanical strength of ceramics [58, 59].

Another technique which has also been proposed as a method to restore the gloss and surface smoothness of ceramics after adjusting with burs is glazing [22]. However, in agreement with other studies, results showed that the fracture resistance was significantly lower in all groups that were glazed compared to the other methods and the control groups [21,22,34,60]. A layer of residual compressive stress is formed in zirconia with bur adjustments. With glazing, that layer is subjected to sintering at high temperature, causing a reverse transformation from the monoclinic to tetragonal phase ($M \rightarrow T$) and the loss of the protective layer, reducing the fracture resistance [61]. The X-ray diffraction analysis showed an even lower percentage of monoclinic phase and decrease in depth-of-processing (compressive stress layer) in groups after LTD.

In addition, glazed groups presented a greater roughness than the rubber-polished groups. Glazing also seem to contribute the adherence of biofilm [14] in conventional ceramics. This technique does not completely correct surface defects caused by the adjustments with diamond burs, as evidenced in SEM images and profilometry analysis. Thus, the protocol Burs + Glazing should be avoided. In summary, for conventional zirconia, polishing after bur adjustments or using the sequence of rubber polishers alone seems to be the best protocol since these protocols produce smoother surfaces and better fracture resistance.

A small Weibull modulus indicates that the distribution of defects is not uniform in a specimen, increasing the probability of failure and reducing the structural reliability of the material [61]. Weibull analysis of the studied groups showed that the UT groups, in general, presented statistically lower Weibull modulus values compared to the conventional zirconia group, similar to other studies [27]. Values were even lower for UT zirconia groups subjected to LTD, indicating that the hydrothermal degradation reduces the structural

reliability of the specimen, although the flexural strength test indicated an increase in fracture resistance after LTD.

The surface morphology analysis showed that the crystals of the conventional zirconia were smaller than the UT zirconia [35]. This characteristic is of great relevance when evaluating the surface roughness of a material, because the smaller the grains, the smoother the surface becomes [62]. However, in this study, the type of zirconia did not influence the roughness of the material. On the other hand, studies indicate that the polishing system influences surface roughness. Polishing systems specified for zirconia provide better results [63,19] than regular systems because they contain a primary abrasive that is effective for the high hardness of the zirconia, which mainly includes diamonds and others abrasives such as SiC, Al_2O_3, [64].

Future investigations should evaluate other finishing and polishing protocols and test configurations. Moreover, regardless of the importance of basic *in vitro* studies, the final decision about adjustment procedures for zirconia restorations should be based on results of randomized controlled clinical trials.

5. CONCLUSION

Based on the results, it seems that the use of diamond polyurethane rubber polishers is the most suitable method for adjusting monolithic zirconia restorations. Glazing after adjustments with diamond burs should be avoided, since it significantly reduces the fracture resistance of these materials.

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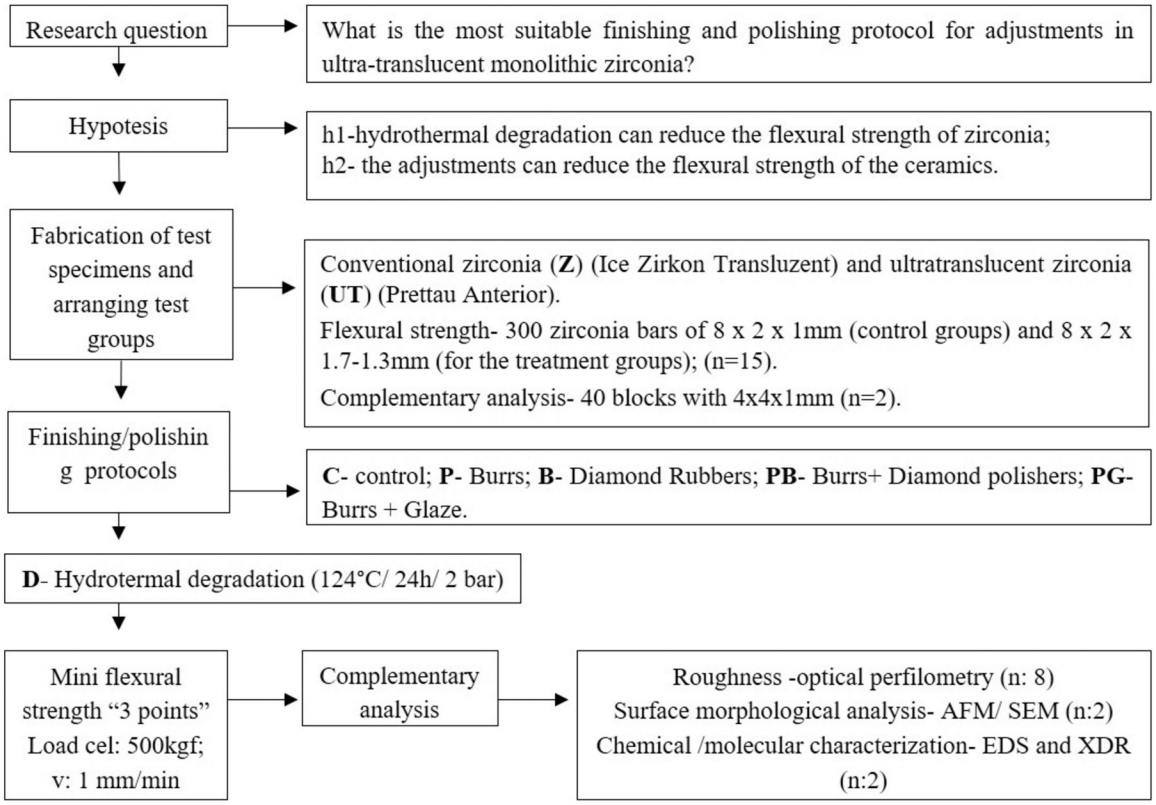


Figure 1.
Flowchart of the study design. D- Hydrothermal degradation.

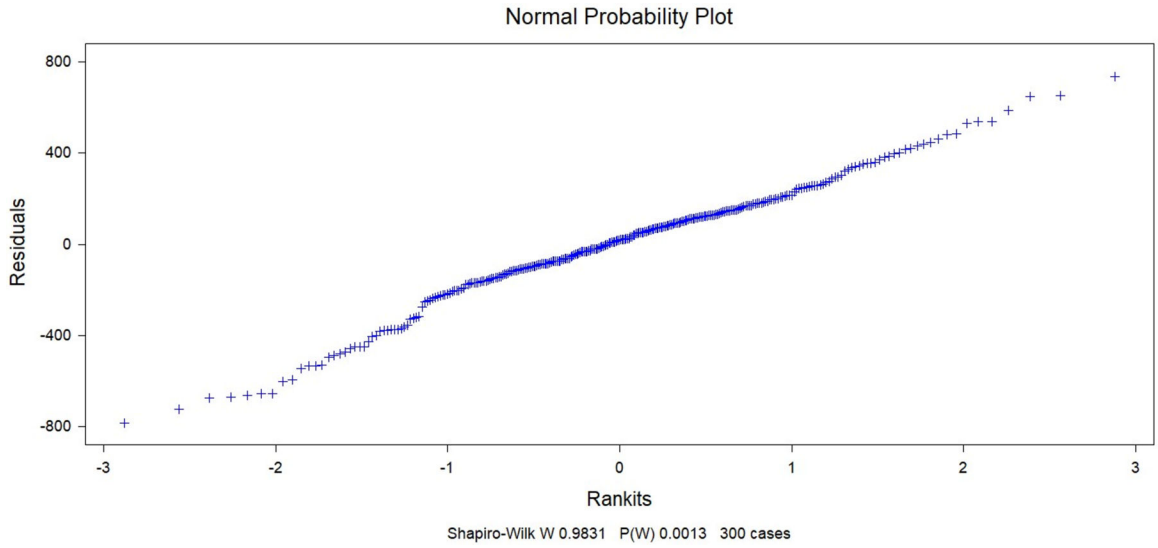


Figure 2.
Normality plot for flexural strength data.

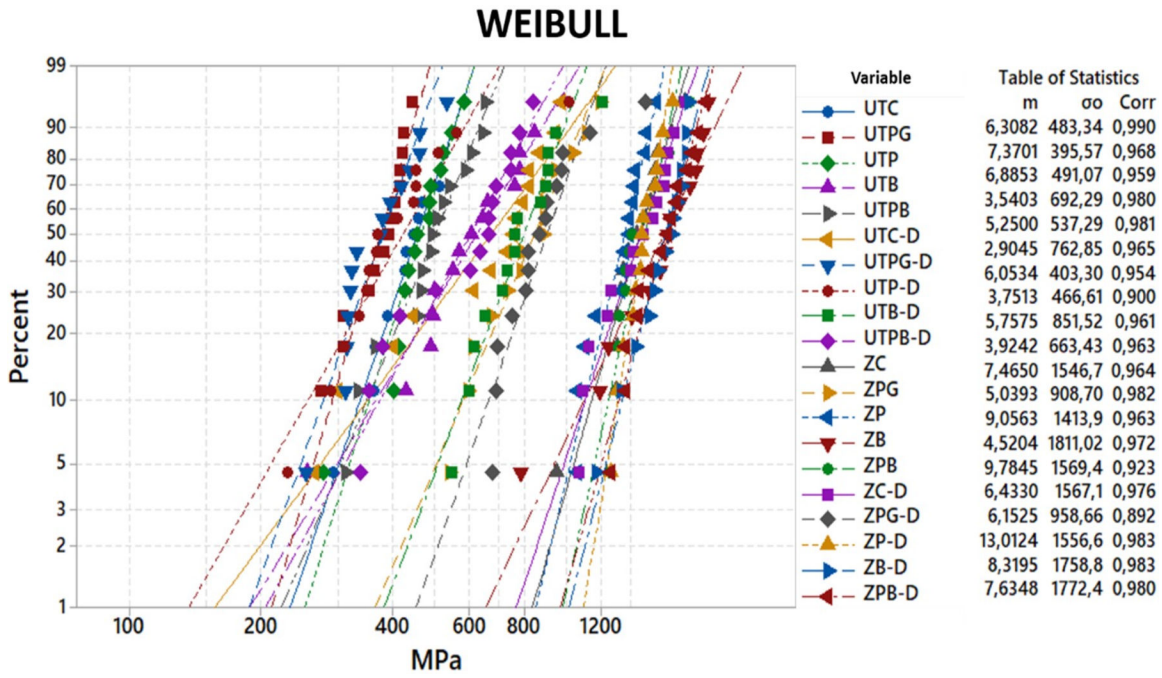
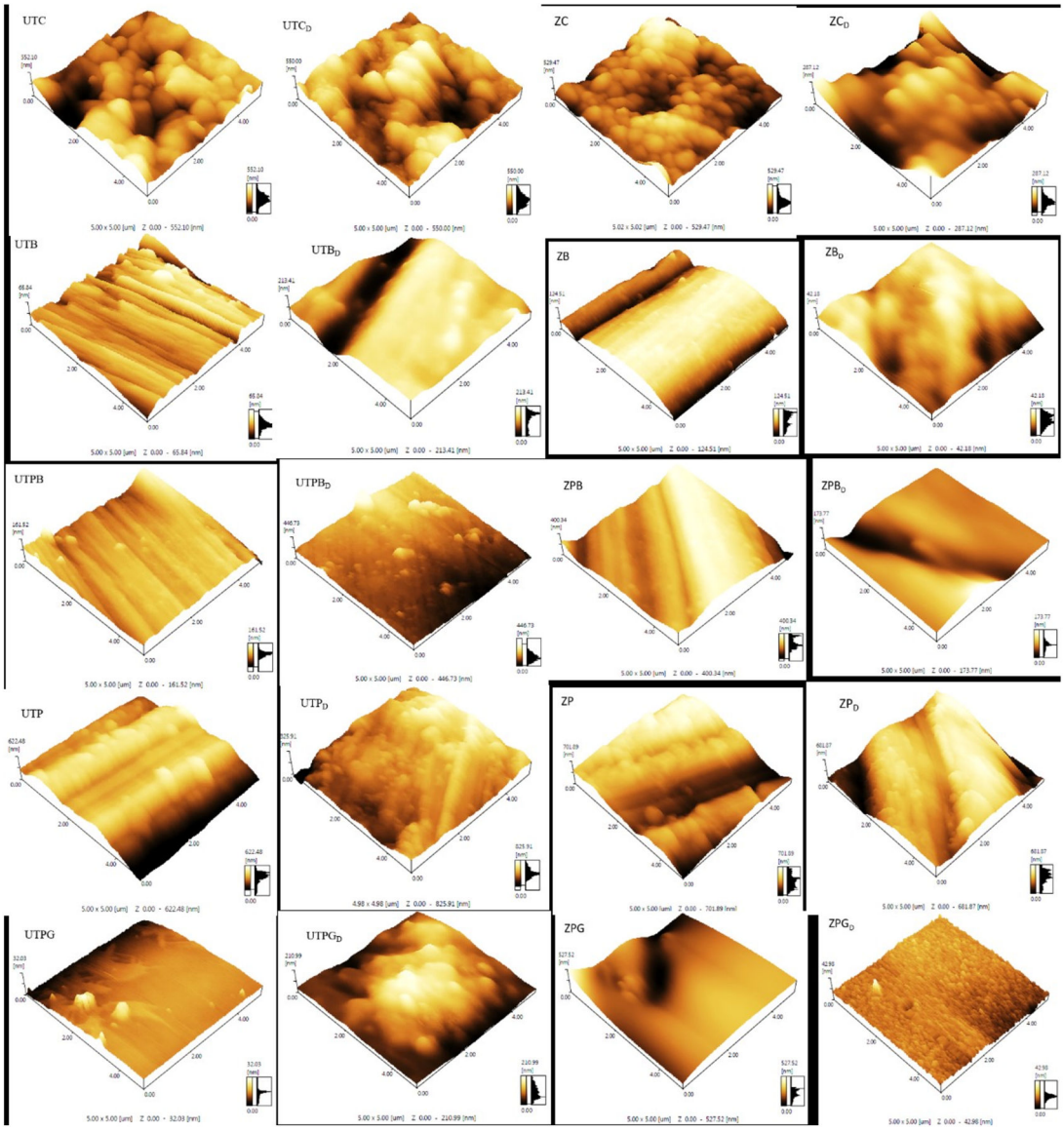


Figure 3.
Weibull plot for flexural strength.



Figures 4.

Micrographs of atomic force microscopy (5μm × 5μm), representing different groups of zirconia.

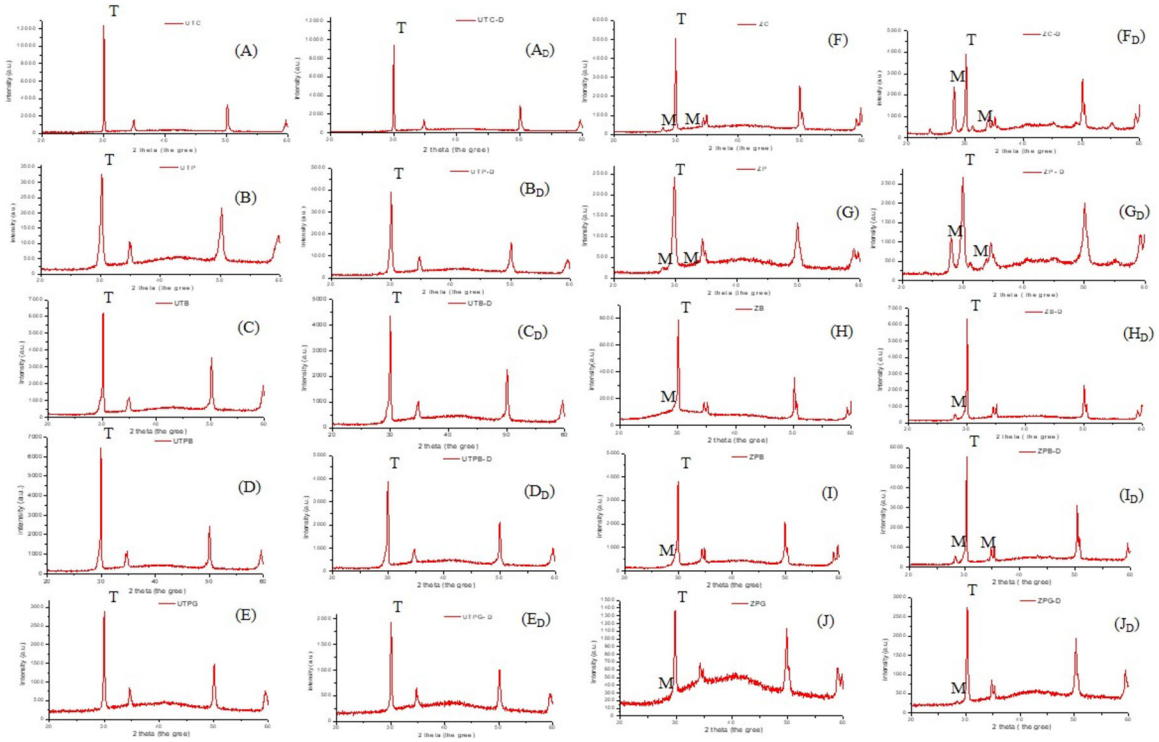


Figure 5.

XRD spectra of zirconia specimens according to experimental groups. A-E, ultra-translucent zirconia with different finishing and polishing protocols. F-J, conventional zirconia with different finishing and polishing protocol. A_D-J_D, zirconia samples after low temperature degradation.

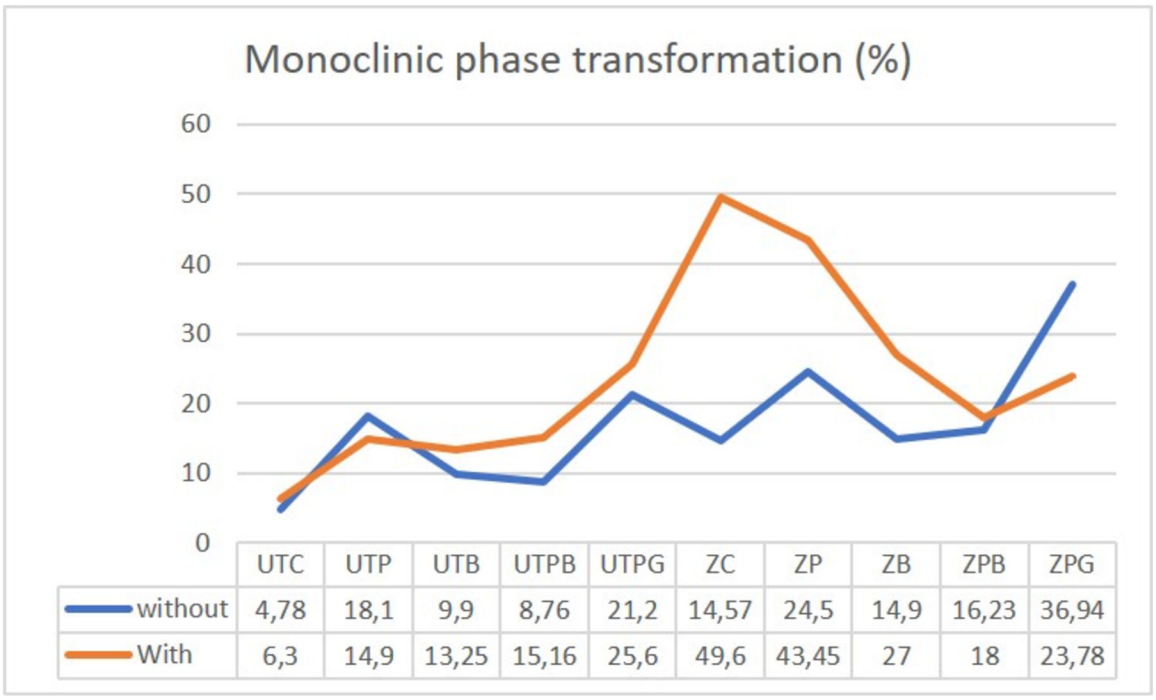
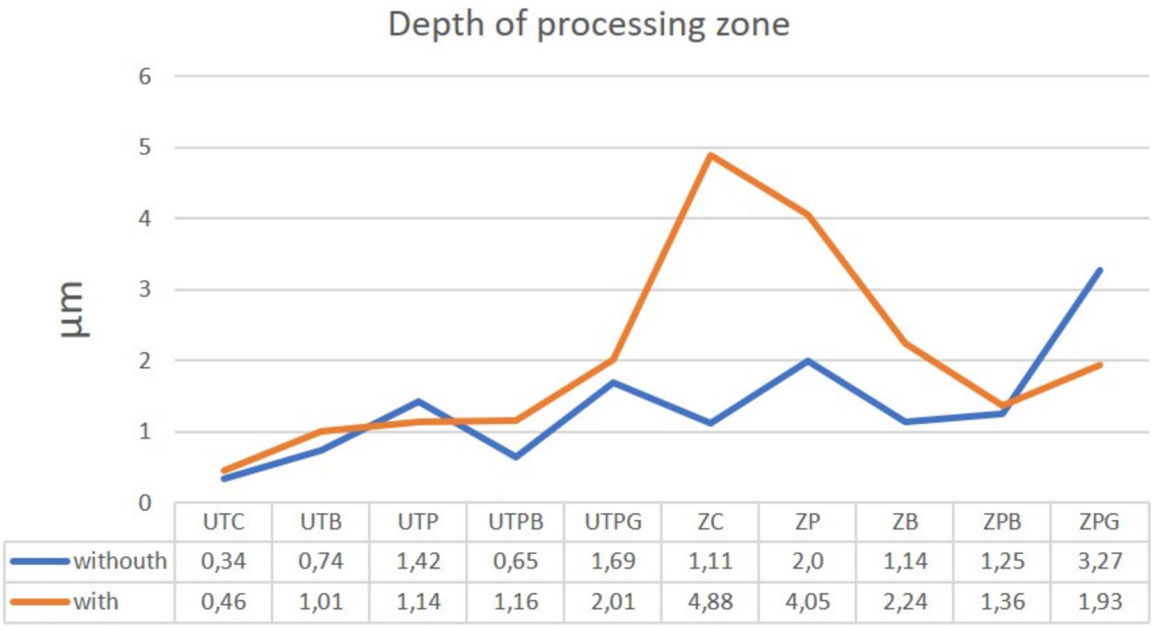


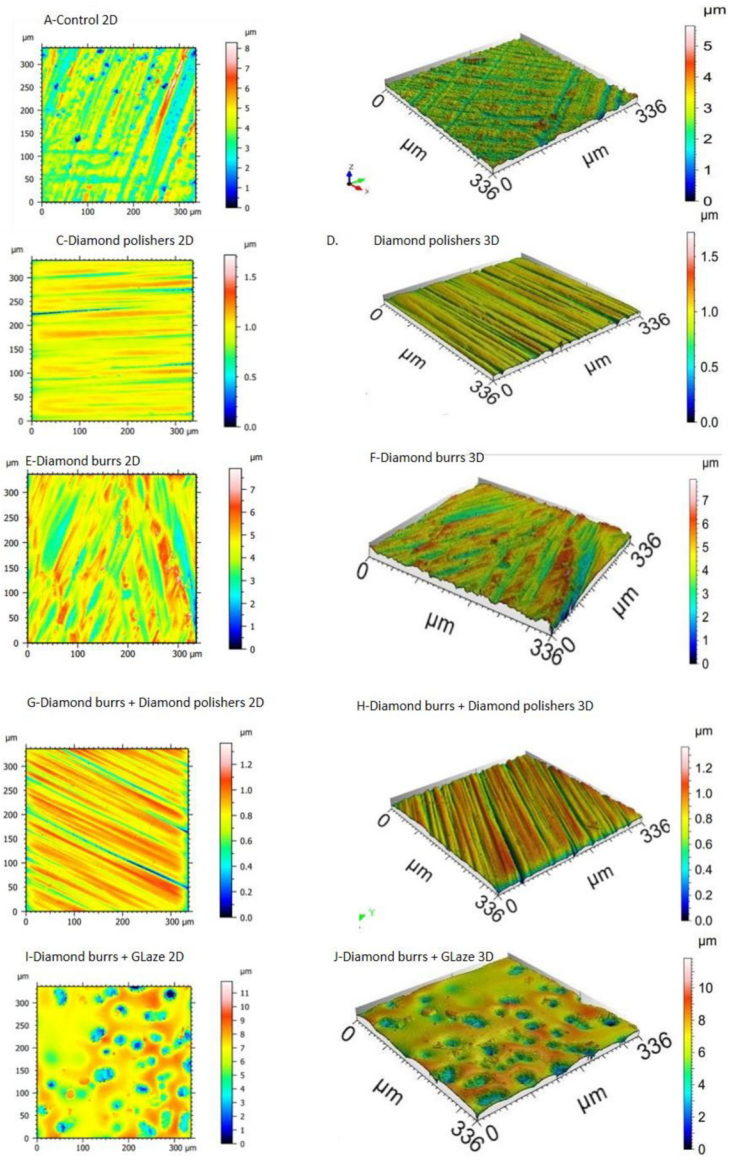
Figure 6.
Monoclinic phase content (%) for all experimental conditions, with and without low temperature degradation.



	UTC	UTB	UTP	UTPB	UTPG	ZC	ZP	ZB	ZPB	ZPG
withouth	0,34	0,74	1,42	0,65	1,69	1,11	2,0	1,14	1,25	3,27
with	0,46	1,01	1,14	1,16	2,01	4,88	4,05	2,24	1,36	1,93

Figure 7.

Depth of the processing zone (µm) for all experimental conditions, with and without low temperature degradation.



Figures 8.

2D and 3D surface topography images of the specimens subjected to different finishing and polishing regimes.

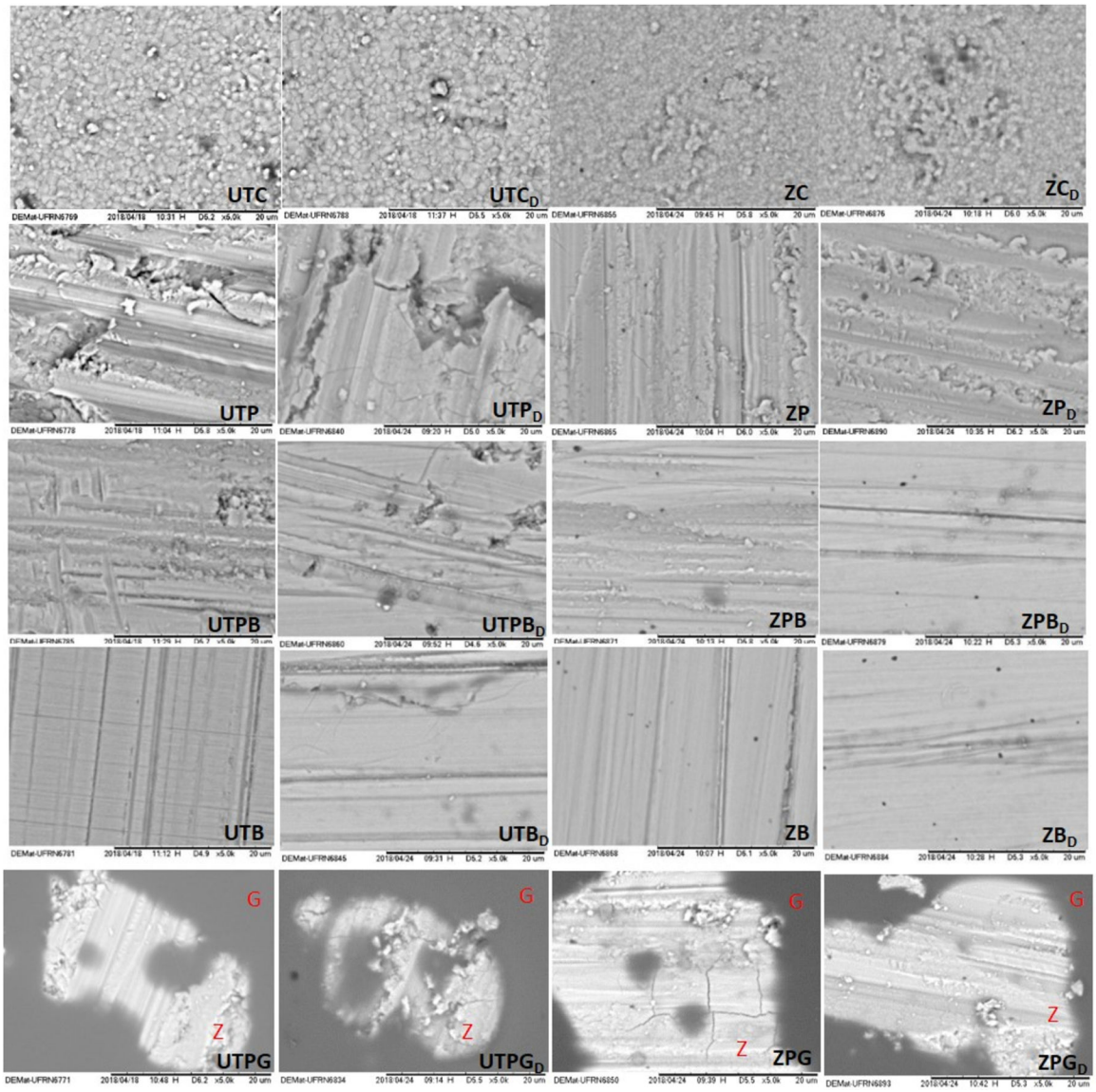


Figure 9.

Scanning electron micrographs at 5,000x magnification, representing different groups of zirconia. G: glaze; Z: Zirconia.

Table 1 -

List of materials used in the study. Material, Trade name, Manufacturer, Composition, Batch number.

Material	Trade name	Manufacturer	Composition	Batch number
Polycrystalline tetragonal zirconia (opaque conventional)	Ice Zirkon transluzent	Zirkonzahn, Gais, Itália.	ZrO_2 > 85%	ZB4224A
			Y_2O_3 - 4 – 6 %	ZB4222E
			Al_2O_3 < 1 %	ZB5302G
			SiO_2 max. 0.02 %	
			Fe_2O_3 max. 0.01 %	
			Na_2O max. 0.04 %	
Polycrystalline tetragonal zirconia full stabilized by yttria (ultratransluzent)	Prettau Anterior	Zirkonzahn, Gais, Italy.	ZrO_2 > 85%	ZB4235E
			Y_2O_3 <12%	ZB5121B
			Al_2O_3 < 1 %	ZB7052D
			SiO_2 max. 0.02 %	ZB5134A
			Fe_2O_3 max. 0.02 %	ZB4235C
				ZB4172F
Glaze	Ivocolor flúor	Ivoclar, Schaan, Liechtenstein	Glass of aluminum silicate of sodium solvents	V35797
Cylindrical Diamond burs #4135	KG Sorensen	KG Sorensen, Cotia, Brazil.	Diamond particles	039910
Diamond Rubber for zirconia finishing and polishing	premium compact kit	Dhpro, Paraná, Brazil.	Polyurethane rubber impregnated with diamond particles	OP166

Table 2 -

Three-way ANOVA results for flexural strength data.

Source	gl	SQ	QM	F	P
Degradation	1	584677	584677	15,95	0,0001 **
Finishing and Polishing	4	1.077E+07	2694233	73,48	0,0000 **
Zirconia	1	5.680E+07	5.680E+07	1548.93	0,0000 **
Degradation × Finishing and Polishing	4	109371	27342.7	0,75	0,5616
Degradation × zirconia	1	3589.68	3589.68	0,10	0,7546
Finishing and Polishing × Zirconia	4	2808281	702070	19,15	0,0000 **
Degradation × Finishing and Polishing × Zirconia	4	432872	108218	2,95	0,0206 **
Error	280	1.026E+07	36668.0		
Total	299	8.178E+07			

* DF: degree of freedom; SS: sum of square; QM: mean square; f: *F*-statistic;

** significant statistic ($p < 0.05$).

Table 3 -

Mean flexural strength (MPa) with standard deviation of experimental groups.

Finishing/polishing	Conventional (Z)		Ultratransluzent (UT)	
	without	with	without	with
Control (C)	1456.1±220.5^{AB}	1463.9±258.2^{AB}	450.8±79.5^{EFG}	678.7±225.4^{CDE}
Burs (P)	1343.1±169.7^{B}	1499.3±133.9^{AB}	460.3±72.8^{EFG}	429.9±182.3^{FG}
Diamond rubber polishers (B)	1654.7±367.7^{A}	1663.5±216.8^{A}	623.5±185.9^{DEF}	791.8±169.4^{CD}
Burs + Diamond Rubber polishers	1497.1±196.0^{AB}	1670.2±252.7^{A}	496.0±103.6^{EFG}	602.3±163.9^{DEFG}
Burs + Glaze	837.3±189.8^{CD}	898.2±212.4^{C}	372.1±56.2^{G}	376.0±75.6^{FG}

*Tukey's test ($p<0,05$). Different letters show statistical differences between groups.

Table 4 -

Characteristic strength (σ_0), Weibull modulus (m), and their respective CI for each group.

Groups	m	CI	σ_0	CI
UTC	6.3^{ABC}	4.1–9.7	483.3	444.1–526.0
UTP	6.8^{ABC}	4.3–11.0	491.0	454.4–530.6
UTB	3.5^{AD}	2.3– 5.5	692.3	595.4– 804.8
UTPB	5.2^{ABC}	3.3–8.3	537.2	485.4– 594.6
UTPG	7.4^{ABC}	4.2– 12.9	395.5	368.0– 425.1
UTC$_D$	2.9^{A}	1.5–5.4	762.8	634.8–916.7
UTP$_D$	3.8^{A}	3.1–4.1	466.6	400.5–543.5
UTB$_D$	5.7^{ABC}	4.3–7.6	851.5	774.6–936.0
UTPB$_D$	3.9^{A}	2.3–6.6	663.4	579.4–759.5
UTPG$_D$	6.0^{ABC}	4.3–8.4	403.3	368.7–441.0
ZC	7.4^{ABC}	5–11.1	1546.7	1440.0–1661.4
ZP	9.0^{ABC}	5.6–14.5	1413.9	1333.3–1499.4
ZB	4.5^{ABC}	2.3–8.6	1811.0	1607.5–2040.3
ZPB	4.5^{B}	2.3–8.6	1811.0	1607.5–2040.3
ZPG	5.0^{ABC}	3.4–7.4	908.7	817.1–1010.5
ZC$_D$	6.4^{ABC}	4.0–10.1	1567.1	1442.6–1702.4
ZP$_D$	13.0^{BC}	9.0–18.7	1556.6	1493.7–1622.1
ZB$_D$	8.3^{ABC}	4.4–15.6	1758.8	1649.0–1876.0
ZPB$_D$	7.6^{ABC}	5.0–11.4	1772.4	1652.5–1901.0
ZPG$_D$	6.1^{BCD}	5.0–7.4	958.6	873.5–1052.0

*Different letters show statistical differences between groups.

Table 5 -

Three-way ANOVA results for roughness data of the experimental groups.

Source	gl	SQ	QM	F	P
Finishing and polishing	4	6.6252	1.65629	64.62	0.0000**
Degradation	1	0.0185	0.01849	0.72	0.3972
Zirconia	1	0.0372	0.03721	1.45	0.2303
Degradation × Finishing and polishing	4	0.1833	0.04582	1.79	0.1347
Finishing and polishing × Zirconia	4	0.0381	0.00953	0.37	0.8284
Degradation × zirconia	1	0.0297	0.02970	1.16	0.2836
Degradation × Finishing and polishing × Zirconia	4	0.1684	0.04211	1.64	0.1669
Error	140	3.5886	0.02563		
Total	159	10.6890			

*DF: degree of freedom; SS: sum of square; QM: mean square; f: *F*-statistic;

**significant statistic ($p < 0.05$).

Table 6 -

Mean roughness (Ra) values (μm) with standard deviations for various experimental groups.

Finishing and Polishing	Ra (μm)
Burs	0.5360 [A] ± 0.15
Burs + Glaze	0.3948 [B] ±0.30
Control	0.3419 [B] ±0.12
Burs+ Diamond rubber polishers	0.0508 [C] ±0.03
Diamond rubber polishers	0.0356 [C] ±0.03

* Different letters show statistical differences between groups.