Original Research
Prosthesis

Effect of grinding and heat treatment on the mechanical behavior of zirconia ceramic

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Declaration of Interests: The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

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DOI: 10.1590/1807-3107BOR-2016.vol30.0012

Submitted: Jan 25, 2015
Accepted for publication: Jun 19, 2015
Last revision: Aug 27, 2015

Abstract: The present study investigated the effect of grinding on roughness, flexural strength, and reliability of a zirconia ceramic before and after heat treatment. Seven groups were tested (n = 15): a control group (labeled CG, untreated), and six groups of samples ground with diamond discs, simulating diamond burs, with grits of 200 µm (G80); 160 µm (G120), and 25 µm (G600), either untreated or heat-treated at 1200°C for 2 h (labeled A). Yttria tetragonal zirconia polycrystal discs were manufactured, ground, and submitted to roughness and crystalline phase analyses before the biaxial flexural strength test. There was no correlation between roughness (R_a and $R_{z)}$) and flexural strength. The reliability of the materials was not affected by grinding or heat treatment, but the characteristic strength was higher after abrasion with diamond discs, irrespective of grit size. The X-ray diffraction data showed that grinding leads to a higher monoclinic (m) phase content, whereas heat treatment produces reverse transformation, leading to a fraction of m-phase in ground samples similar to that observed in the control group. However, after heat treatment, only the G80A samples presented strength similar to that of the control group, while the other groups showed higher strength values. When zirconia pieces must be adjusted for clinical use, a smoother surface can be obtained by employing finer-grit diamond burs. Moreover, when the amount of monoclinic phase is related to the degradation of zirconia, the laboratory heat treatment of ground pieces is indicated for the reverse transformation of zirconia crystals.

Keywords: Ceramics; Dental Alloys; Dental Materials.

Introduction

Advances in CAD/CAM systems have enabled the optimization of ceramic materials, extending their clinical applications. Yttria tetragonal zirconia polycrystal (Y-TZP) stands out among other restorative dental materials due to its high chemical stability and biocompatibility, and superior mechanical properties.[1,2] These highly favorable mechanical properties allow for the application of all-ceramic Y-TZP crowns and bridges in the posterior region, also reducing the amount of tooth structure removal during preparation, since a smaller thickness of the restorative material is required. These characteristics are very attractive for applications in dental prostheses, in which strength and esthetics requirements are crucial[3] and/or there is a narrower interocclusal space.

Zirconia is a polymorphic material existing in three different crystalline forms, stabilized in tetragonal phase at room temperature. Yttrium oxide proved to be an excellent option in this context, since it creates a fine-grain microstructure (3Y-TZP).[1] Nevertheless, the tetragonal to monoclinic (t→m) phase transformation may be triggered by different stimuli, such as stress concentration or low-temperature aging, in the presence of humidity, leading to low-temperature degradation.[4]

Despite the advances in CAD/CAM manufacturing systems, and pre-sintered ceramic blocks, fine adjustments of already sintered zirconia surfaces are still performed by dental technicians or clinicians,[5] in some cases, for a better fitting of a prosthetic restoration. These manual procedures could trigger the t→m phase transformation and grinding, creating a compressive stress layer, due to an increase in the volume of the material (zirconia) by approximately 3%, in addition to producing some surface defects in the material. When the depth of these defects is greater than the thickness of the compressive layer, the defects can act as stress concentration zones, which can impair the mechanical properties.[6,7,8] Nevertheless, when the depth of these defects is lower than the thickness of the compressive stress layer, crack propagation is hindered and catastrophic failures are avoided by the surrounding compressive stresses.[9,10] However, grinding can contribute to premature aging, causing the material to lose its ability to prevent crack propagation.[11]

A strong correlation has been reported in the literature between the amount of monoclinic phase and degradation of the mechanical properties of zirconia.[12,13] Therefore, procedures that prevent t→m phase transformation or that cause reverse m→t transformation should be investigated as alternative ways to prevent the degradation of mechanical properties. Different heat treatment protocols have been proposed to induce surface healing by promoting reverse m→t transformation. These protocols relieve the stress present in the compression layer formed as well as the residual stresses induced on the zirconia surface.[14]

The aim of this study was to assess the effect of diamond grinding of zirconia, with and without heat treatment, on strength and surface characteristics of this ceramic material. The tested hypotheses are that: (1) the grinding of zirconia with larger grit-size devices leads to a decrease in flexural strength and to an increase in monoclinic content and in surface roughness; and (2) heat treatment restores the original characteristics of the material.

Methodology

The materials used in this study are listed in Table 1.

Lava Frame (3M ESPE) is a tetragonal zirconia polycrystal partially stabilized with 3mol-% yttria. This ceramic presents a very fine microstructure, containing fine grains and few slightly large grains (mean diameter of 104.9 nm ± 48.5).[15]

Preparation of specimens

Disc-shaped specimens were manufactured according to ISO 6872/2008.[16] Pre-sintered blocks of Y-TZP (Lava Frame) were ground into cylinders with SiC paper (3M) under water cooling and sectioned with a precision saw (ISOMET 1000, Buehler, Lake Bluff, USA) into slices of 18 mm (Ø) × 1.6 mm (thickness). To remove irregularities left by the cutting tool, the disc surfaces were finely ground with a 1200-grit SiC paper, and the samples were then subjected to firing at 1530°C for 2 h in an especially designed furnace (Zyrcomat T, Vita Zahnfabrik, Bad Säckingen, Germany), according to the manufacturer's instructions. The final disc dimensions were 14 mm × 1.2 mm. The discs were divided into seven groups: CG (control group)- discs without grit blasting and without heat treatment; G80- discs ground with a 200 µm grit paper and without heat treatment; G80A- discs ground with a 200 µm grit paper and heat-treated; G120- discs ground with a 160 µm grit paper and without heat

Table 1. Material, respective manufacturers, city and country.

Material	Manufacture	City and Country
Lava Frame	3M ESPE	Seefeld, Germany
SIC paper (1200 grit)	3M	Saint Paul, USA
Dia-grid Diamond Discs	Allied high tech Products Inc.	Compton, USA

treatment; G120A- discs ground with a 160 µm grit paper and heat-treated; G600- discs ground with a 25 µm grit paper and without heat treatment; and G600A- discs ground with a 25 µm grit paper and heat-treated. Both surface and heat treatments are explained in what follows.

Surface treatment

The samples were attached to a polishing machine (AutoMet 250, Buehler, Lake Bluff, USA) and subjected to abrasion with diamond discs (Dia-Grid Diamond Discs, Allied High Tech Products) for 60 s using a 60 N load, at 300 rpm on the turntable (clockwise) and at 40 rpm on the table top device (counter-clockwise), under constant water cooling, with one of the following grit sizes: 80 (200 µm), 120 (160 µm) or 600 (25 µm). These grit sizes were selected according to Kim *et al.*,[10] to simulate super-coarse, coarse, and extra-fine diamond burs, respectively. Half of the ground specimens were then heat-treated at 1200°C for 2 h to relieve the residual stresses caused by grinding and to promote reverse m→t transformation.[14] Specimens not subjected to abrasion or heat treatment were used as control (n = 15).

Micromorphological and surface roughness analysis

The surface roughness of each specimen was analyzed using a surface roughness tester (SJ-400, Mitutoyo, Kanagawa, Japan). Two perpendicular measurements were performed for each specimen. The arithmetic mean roughness (R_a, µm) and the maximum profile height (R_z, µm) were measured and correlated with the strength data. For the qualitative determination of the micromorphological pattern generated by grinding, the specimens were analyzed under a scanning electron microscope (SEM) (n = 2, JSM-6360, JEOL, Tokyo, Japan).

X-ray diffraction analysis of phase transformations

Phase transformations were observed by measuring the monoclinic phase fraction in each sample (n = 2) using X-ray diffraction (XRD). The data were collected with a 2θ diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) using Cu-K_α radiation, in a 2θ range of 20° to 65°, with step sizes of 0.5 s and 0.03. The monoclinic phase fraction was calculated using the Garvie and Nicholson's method.[17]

Biaxial flexural strength

The samples were subjected to a biaxial flexural strength test according to ISO 6872/2008,[16] in a universal testing machine (EMIC, São José dos Pinhais, Brazil). Disc-shaped specimens were positioned with the treated surface face down (tensile stress) on three support balls (Ø = 3.2 mm) placed 10 mm apart from each other in a triangular arrangement. The assembly was immersed in water, and a flat circular tungsten piston (Ø = 1.6 mm) was used to apply an increasing load (1 mm/min) until catastrophic failure. Before the test, adhesive tape (12 mm × 10 mm, 3M ESPE) was fixed on the compressive side of the discs to retain the fragments[18] and enhance the contact between piston and sample.[19] The strength σ (MPa) was calculated according to ISO 6872/2008.[16] After the test, the specimens were analyzed under a stereomicroscope (SteREO Discovery. V12, Carl Zeiss) to verify the location of the fracture origin.

Data analysis

One-way analysis of variance and post-hoc Tukey's test were used to analyze the roughness and biaxial flexural strength values ($\alpha < 0.05$). Roughness and strength values were evaluated as to correlation (Pearson's correlation) (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, USA). The latter were subjected to Weibull analysis[20] to determine the characteristic strength (*i.e.*, the strength at a 63.21% failure probability, which represents a more objective measure of strength than its average value), and the reliability of the tested material, based on the Weibull modulus to quantify the distribution of the strengths of the different samples. Weibull parameters were calculated according to DINENV843-5:1997-01.[21]

Results

No correlation was found between roughness parameters and strength values (Ra, $r^2 = 0.0303$, $p = 0.7587$; Rz, $r^2 = 0.0226$, $p = 0.8189$). The roughness data (mean Ra and Rz values) are shown in Table 2;

the grit size of diamond discs affected both the Ra ($p < 0.001$) and Rz parameters ($p < 0.001$).

Representative micrographs (Figure 1) show that surface treatment altered the micromorphological pattern after grinding, whereas heat treatment did not induce significant alterations in this pattern. The roughness analysis highlights a lower roughness for G600 (smaller granulation) than G80, G120, and GC groups, whereas heat treatment did not lead to significant alterations (Table 2).

X-ray diffraction data (Figure 2) show that grinding promoted a higher m-phase content than the control group, and that heat treatment induced the reverse transformation of the monoclinic phase, achieving a m-phase content similar to that of the control group.

Mean flexural strength and the respective standard deviations are described in Table 3. The control group presented the lowest values of flexural strength; groups without heat treatment and G120A were statistically different from the control group. The Weibull modulus (m) was not affected by abrasion or heat treatment. The characteristic strength (σ_c) was higher after abrasion with diamond discs, irrespective of their grit size. After heat treatment, only G80A presented strength similar to that of the control group, while the other groups sustained higher strength (Table 3).

The fracture origin was always located on the ceramic surface and no internal defects were found. Figure 3 shows a representative image of the common fracture origin found in all tested groups.

Discussion

The present work evaluated the effect of grinding on surface roughness and mechanical strength. The efficacy of post-grinding heat treatments to induce reverse t-m phase transformation was also investigated. Contrary to the initial hypotheses, surface roughness did not directly influence flexural strength. Based on the R_a values, only samples ground with a 600-grit diamond disc (corresponding to extra-fine diamond burs[10]) presented lower surface roughness, regardless of the heat treatment used. In the other test groups, roughness was similar to that of the control group. Even though no clear relationship was identified between biaxial flexural strength and surface roughness, as corroborated by Sato *et al.*,[22] abrasion/polishing may be responsible for the elimination of deeper scratches produced during the manufacturing

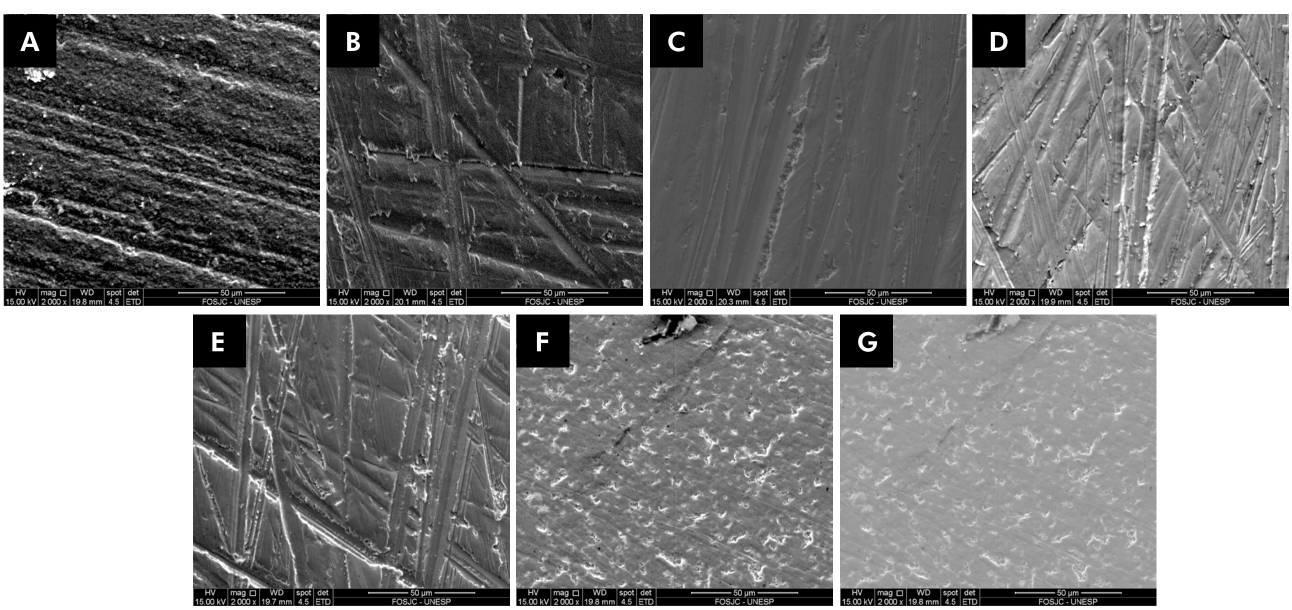

Figure 1. Scanning electron microscopy (2,000x magnification) of surface patterns in each group. Control (Top, center); G80 (1st row, left column); G80A (1st row, right column); G120 (2nd row, left column); G120A (2nd row, right column); G600 (3rd row, left column); G600A (3rd row, right column).

Table 2. Surface roughness values of the test groups (mean and standard deviation, in μm).

Group	Ra	Rz
GC	0.28 (0.06)[a]	2.22 (0.42)[a]
G80	0.29 (0.03)[a]	2.06 (0.30)[ab]
G80A	0.26 (0.03)[a]	1.83 (0.25)[b]
G120	0.29 (0.03)[a]	2.31 (0.34)[a]
G120A	0.31 (0.04)[a]	2.19 (0.37)[a]
G600	0.11 (0.02)[b]	0.77 (0.18)[c]
G600A	0.12 (0.05)[b]	0.88 (0.31)[c]

*The same superscript letters in the respective columns indicate statistical similarity ($\alpha < 0.05$).

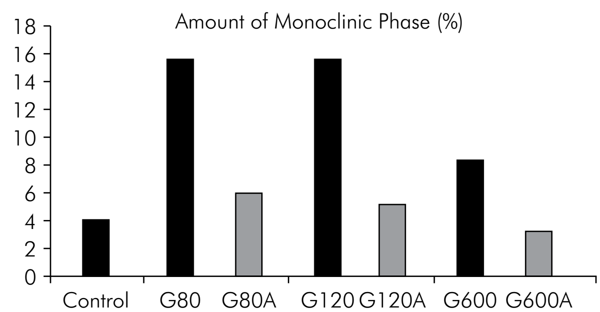

Figure 2. Relative amount of monoclinic phase, according to X-ray diffraction analysis in each tested group: CG (4.15%), G80 (15.64%), G80A (5.97%), G120 (15.6%), G120A (5.24%), G600 (8.39%), G600A (3.27%).

process. Polishing/abrasion eliminates a thin layer of material, also eliminating surface defects and increasing strength.[23]

Surface roughness plays a crucial role in the resistance of ceramics,[24] usually showing a significant and negative correlation with flexural strength (higher roughness with lower flexural strength)[25] unlike the results of the present study. In addition to roughness and the amount of monoclinic phase, other factors may have influenced the flexural strength of Y-TZP discs. Abrasion/polishing may also produce deeper scratches, depending on the initial conditions of the material surface and also on the abrasion/polishing protocols. As observed in Figure 1, specimens abraded with 80- and 120-grit discs presented apparently larger and deeper scratches than those in the control group. The Y-TZP samples from the present study were polished with 1200-gritpaper before sintering. The pattern generated by the CAM milling unit was not simulated.

The influence of abrasion on the flexural strength of zirconia ceramics is uncertain. Several factors, such as roughness, plastic deformation, and the volume percentage of transformed zirconia are implicated. The last-mentioned factor depends on the rate of t→m phase transformation, on abrasion severity, and on local temperatures. [2,6,26]

After grinding, the flexural strength of zirconia discs was higher than that of the control group. This is probably due to the elimination of deeper flaws and to a more homogenous surface after grinding, and also to an increase in the fracture toughness of Y-TZP due to t→m phase transformation (Figure 2).[27] The surface pattern was probably standardized after grinding in G120 and G600, with or without heat treatment (Figure 1), leading to a more homogeneous stress distribution and to higher characteristic strength values when compared to the other groups (Table 3). Finer grit instruments are supposed to present, besides smaller grains, a higher number of grains and a lower distance between them, causing scratches that are greater in number and closer to each other (Figure 1), creating a more homogeneous surface than in the case of separated few scratches, probably present in G80. This last-mentioned group presented a higher characteristic strength than that of the control group, which is probably associated with the higher amount of monoclinic phase (compression layer). The characteristic strength of G80A was similar to that of the control group; heat treatment reduced the monoclinic phase content in this group (Figure 2), leading to lower flexural strength.

Heat treatment did not affect the characteristic strength in the groups in which a disc with the same grit size was used (Table 3). The enhanced strength of Y-TZP ceramic after finer abrasion with diamond devices (G120 and G600) had been reported previously,[28] while coarser abrasion resulted in strength reduction. Moreover, a compressive stress may still be present on the surface in these groups, even after reverse phase transformation promoted by heat treatment.

The transformation rate could also be related to the zirconia grain size: larger tetragonal grains result in lower phase stability.[29] Lava™ ceramic exhibited a large grain size, as previously reported,[30]

Table 3. Mean flexural strength (FS), standard deviation (SD), characteristic strength (σc), Weibull modulus (m), and the respective confidence intervals (CI) for each group.

	FS (MPa) and SD*	σ_c	CI (95%)**	m	CI (95%)**
GC	817.18 (±115.2)^{C}	863.87	815.87–914.69	9.29	6.14–14.0
G80	995.30 (±104.6)^{AB}	1039.7	996.90–1084.3	12.3	8.30–18.2
G80A	891.70 (±150.0)^{BC}	952.96	886.78–1024.1	7.40	4.89–11.2
G120	1019.3 (±182.4)^{AB}	1090.8	1009.7–1178.3	6.89	4.63–10.2
G120A	1070.8 (±130.8)^{A}	1117.9	1069.2–1168.8	11.5	7.72–17.2
G600	991.45 (±121.7)^{AB}	1054.0	998.10–1112.9	9.51	6.50–13.9
G600A	941.85 (±156.6)^{ABC}	1005.4	934.27–1081.9	7.27	4.87–10.8

*Different letters indicate statistical difference (p = 0.000).
**Statistically different values do not present overlapping confidence intervals.

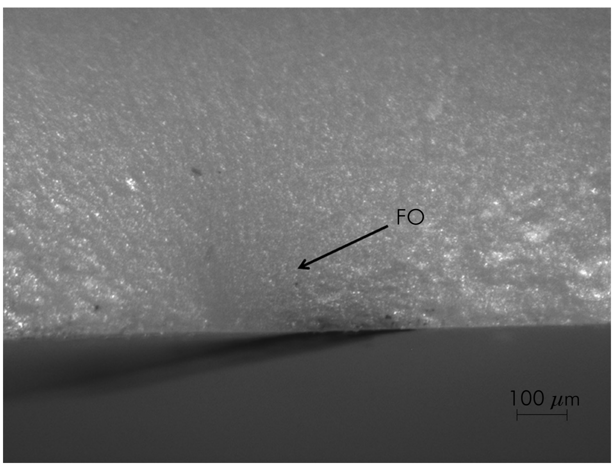

Figure 3. Representative image (90x magnification) of the fracture origin (FO) located on the surface of the sample (Group 120A).

and thus a corresponding greater probability of phase transformation.[31]

The Weibull modulus of the tested groups was similar, regardless of grit size or heat treatment. This parameter describes the reliability of the material. A higher modulus indicates a small content of flaws and higher reliability.[32] The procedures performed on the test samples, *i.e.*, abrasion and heat treatment, did not produce critical defects.

The emergence of the monoclinic phase and the associated microcracking were found to be the most likely causes for the degradation of mechanical properties of zirconia-based ceramics.[12] Thus, reverse transformations from m- to t-zirconia would be desirable when stress is applied to the ceramic surface before cementation of the prosthesis. The heat treatment applied here (1200°C / 2 h)[14] was successful in bringing the monoclinic content after abrasion back to baseline values, as shown in Figure 2. The m→t phase transformation was also observed after heat treatment for a shorter time (1200°C / 10 min),[22] at a lower temperature (900°C / 1 h)[2], or both (930°C and 910°C / 1 min)[7], showing that transformation may occur after a given temperature is reached, regardless of the treatment time. The protocol applied in this study is known to relieve residual stresses without repairing cracks and/or scratches.[14,33] As shown in Table 2, surface roughness was not affected by heat treatment.

Preis *et al.*[11] investigated roughness and phase transformation (t→m) of Y-TZP after dental adjustment procedures, concluding that polishing must be carefully performed to obtain a smooth surface, with a low amount of monoclinic phase. Pereira *et al.*[34] also found higher flexural strength values for Lava Frame Y-TZP when discs were abraded with finer grit instruments. The polishing/abrasion performed in this study (diamond discs attached to a polishing machine) was controlled for direction and applied load. This may lead to different damage when compared to clinical adjustments, which are not under any kind of load or direction control. For this reason, the protocol used in this study was probably less deleterious to the material properties than the ones used by clinicians.

Clinically, the adjustment should be performed with fine diamond burs to avoid the decrease of mechanical properties and, consequently, the premature failure of all-ceramic restorations. Further investigations are needed to evaluate the effects of abrasion and heat treatment, as well as of the amount of monoclinic phase, on the long-term mechanical behavior of zirconia.

Conclusion

When the clinical fine adjustment of zirconia restorations is necessary, it should be performed with finer grit diamond burs (120 and 600 grit sizes, corresponding to coarse and extra-fine diamond burs), thus not affecting the efficacy of the restorations. Heat treatment can be applied after the adjustment when absence of the monoclinic phase is desirable. The heat treatment protocol (1200°C / 2 h) employed in this work proved to be effective.

Acknowledgments

The authors thank the Research Foundation of the State of São Paulo (*Fundação de Amparo à Pesquisa do Estado de São Paulo* - FAPESP) for the financial support (FAPESP - 2011/16716-0).

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