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Residual compressive surface stress increases the bending strength of dental zirconia

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ABSTRACT

Objective. To assess the influence of surface treatment and thermal annealing on the four-point bending strength of two ground dental zirconia grades.

Methods. Fully-sintered zirconia specimens ($4.0 \times 3.0 \times 45.0 \text{ mm}^3$) of Y-TZP zirconia (LAVA Plus, 3M ESPE) and Y-TZP/ Al_2O_3 zirconia (ZirTough, Kuraray Noritake) were subjected to four surface treatments: (1) 'GROUND': all surfaces were ground with a diamond-coated grinding wheel on a grinding machine; (2) 'GROUND + HEAT': (1) followed by annealing at 1100°C for 30 min; (3) 'GROUND + Al_2O_3 SANDBLASTED': (1) followed by sandblasting using Al_2O_3 ; (4) 'GROUND + CoJet SANDBLASTED': (1) followed by tribochemical silica (CoJet) sandblasting. Micro-Raman spectroscopy was used to assess the zirconia-phase composition and potentially induced residual stress. The four-point bending strength was measured using a universal material-testing machine.

Results. Weibull analysis revealed a substantially higher Weibull modulus and slightly higher characteristic strength for ZirTough (Kuraray Noritake) than for LAVA Plus (3M ESPE). For both zirconia grades, the 'GROUND' zirconia had the lowest Weibull modulus in combination with a high characteristic strength. Sandblasting hardly changed the bending strength but substantially increased the Weibull modulus of the ground zirconia, whereas a thermal treatment increased the Weibull modulus of both zirconia grades but resulted in a significantly lower bending strength. Micro-Raman analysis revealed a higher residual compressive surface stress that correlated with an increased bending strength.

Significance. Residual compressive surface stress increased the bending strength of dental zirconia. Thermal annealing substantially reduced the bending strength but increased the consistency (reliability) of 'GROUND' zirconia.

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1. Introduction

Today, dental zirconia most commonly consist of yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) based on the TZ-3YE (Tosoh, Tokyo, Japan) starting powder [1]. Recently, more aging-resistant and stronger zirconia, such as Ce-TZP/Al₂O₃ zirconia (NANOZR, Panasonic, Osaka, Japan) or Y-TZP/Al₂O₃ zirconia (ZirTough, Kuraray Noritake, Tokyo, Japan), have been introduced in dentistry [2].

In many cases, the dental technician needs to additionally grind the inside of the zirconia core to improve the fit of the zirconia restoration onto the tooth preparation [3]. Furthermore, a recent systematic review revealed that different mechanical surface pre-treatments are essential for the composite cement to durably bond to zirconia, this in light of an adhesive luting procedure [4]. Among them, aluminium oxide (Al₂O₃) sandblasting or tribochemical silica sandblasting with 30- and 110-μm silica-coated Al₂O₃ particles are quite important to achieve durable bonding to zirconia [5]. However, the surface of the abraded zirconia will be transformed, i.e., constrained as well as damaged; this stress-induced transformation may influence the long-term clinical performance of zirconia restorations [3].

Several papers reported on the influence of different surface treatments on the mechanical properties of dental zirconia. However, no consensus was reached in literature whether surface treatments are detrimental for the mechanical properties of dental zirconia. Some of those studies reported an increased bending strength of zirconia [6–14], whereas others reported a decreased strength as result of surface damage produced by the surface treatments [15–18]. Moreover, the mechanical properties of surface-treated newer zirconia, such as Y-TZP/Al₂O₃ zirconia (and Ce-TZP/Al₂O₃ zirconia, however not investigated in this study) have not much been studied yet. The objective of this study was therefore to evaluate the influence of different surface treatments on the bending strength of two different, i.e., Y-TZP and Y-TZP/Al₂O₃, dental zirconia. The null hypothesis tested was that different surface pre-treatments do not influence the bending strength and consistency (reliability) of dental zirconia.

2. Materials and methods

Fully-sintered Y-TZP LAVA Plus (3M ESPE, Seefeld, Germany) zirconia and Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia were provided by the manufacturers in the form of 3.5 × 4.5 × 45.0 mm bending bars. Next, the specimens were ground to the size of 3.0 × 4.0 × 45.0 mm, after which they were assigned to four surface treatments (Table 1): (1) 'GROUND': all surfaces were ground using a polymer-bonded diamond-grinding wheel (D46 SW 50–X2, Technodiamant, Almere, The Netherlands) on a grinding machine (JF415DS, Jung, Göppingen, Germany); (2) 'GROUND + HEAT': (1) followed by annealing at 1100 °C in air for 30 min (Nabertherm, Germany); (3) 'GROUND + Al₂O₃ SANDBLASTED': (1) followed by sandblasting using 50 μm Al₂O₃ particles (Danville Engineering, San Ramon, CA, USA); (4) 'GROUND + CoJet SANDBLASTED': (1) followed by tribochemical silica sandblasting with 30 μm

silica-coated Al₂O₃ particles using CoJet (3M ESPE). All surfaces of the bending bar received the same surface treatment.

One specimen from each surface-treated zirconia was used for micro-structural investigation using scanning electron microscopy (SEM, JSM-6610LV, JEOL, Tokyo, Japan) employing the following conditions (gold coated, 10^{−5} mbar pressure, 15 kV energy range, 85 μA beam current, 1000× magnification, secondary electron image).

Micro-Raman spectroscopy was used to measure the monoclinic zirconia (*m*-ZrO₂) surface-volume fraction, and to assess the nature and extent of potential residual stress on the surface [19]. Depth-profile Raman spectra (SENTERRA, BrukerOptik, Ettlingen, Germany) were also collected from the top 20-μm material of surface-treated zirconia specimens using the following conditions: Ar-ion laser with a wavelength of 532 nm, 20 mW power at sample and 100× objective. The spectrum integration time was 20 s with the recorded spectra averaged over three successive measurements. For each specimen, at least 12 measurements were performed using a pinhole aperture of 50 μm. The *m*-ZrO₂ surface-volume fraction was calculated using the equation proposed by Tabares et al. [20]:

$$V_m = \frac{I_m^{181} + I_m^{190}}{0.32 (I_t^{147} + I_t^{265}) + I_m^{181} + I_m^{190}}$$

where *I_m* and *I_t* represent the height of the intensities of the monoclinic and tetragonal peaks, respectively, and the superscripts refer to the Raman wavenumber in cm^{−1}. In addition, for each measurement, the Raman wavenumber of the tetragonal zirconia (*t*-ZrO₂) band around 147 cm^{−1} was traced to analyze the residual surface-stress level [19] using a curve fitting software (fity 1.2.1, Marcin Wojdyr, Warsaw, Poland) [21].

The bending strength was determined using a four-point bending test, following a modified ISO 6872 standard with an inner span of 20 mm and an outer span of 40 mm. The specimens were loaded at a crosshead speed of 0.1 mm/min until failure in a universal testing machine (Instron 4467, Instron, Bucks, UK). The bending strength results were statistically analyzed using Weibull analysis; pivotal confidence bounds were calculated using Monte Carlo simulation [22]. Different groups were compared at the B63.2 unreliability level, commonly referred to as the characteristic strength, σ₀, i.e., the strength for which the survival probability is 36.8%. All tests were performed at a significance level of α = 0.05 using a software package R3.0 and Abrem (R Foundation for Statistical Computing, Vienna, Austria) and a custom-made script for this software. Additionally, the degree of correlation between the Raman wavenumber of the *t*-ZrO₂ band around 147 cm^{−1} and the bending strength was calculated for each grade using the abovementioned software.

3. Results

Representative SEM photomicrographs of the four surface treatments from each zirconia grade (LAVA Plus, 3M ESPE; ZirTough, Kuraray Noritake) are shown in Fig. 1. For both zirconia grades, the 'GROUND' and 'GROUND + HEAT' treated zirconia revealed a similar microstructure. The 'GROUND + Al₂O₃

Table 1 – Summary of the different mechanical surface treatments.

Surface treatment	Procedure
'GROUND'	All surfaces were ground with a polymer-bonded diamond-grinding wheel (D46 SW 50—X2, Technodiamant, Almere, The Netherlands) on a grinding machine (JF415DS, Jung, Göppingen, Germany).
'GROUND + HEAT'	'GROUND' followed by annealing at 1100 °C in air for 30 min.
'GROUND + Al ₂ O ₃ SANDBLASTED'	'GROUND' followed by sandblasting with 50 µm Al ₂ O ₃ particles (Danville, Danville, CA, USA) for 15 s/cm ² at a distance of 10 mm with an intraoral air-abrasion device (Microetcher, Danville Engineering, Danville, CA, USA).
'GROUND + CoJet SANDBLASTED'	'GROUND' followed by sandblasting with 30 µm silica-coated Al ₂ O ₃ particles (CoJet sand, 3M ESPE, Seefeld, Germany) for 15 s/cm ² at a distance of 10 mm with an intraoral air-abrasion device (CoJet Prep, 3M ESPE, Seefeld, Germany).

SANDBLASTED' zirconia resulted in a rougher surface than the 'GROUND + CoJet SANDBLASTED' zirconia. For 'GROUND' or 'GROUND + HEAT' treated Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia, dark contrast alumina particles were revealed.

All data of the Weibull analysis are graphically presented in Fig. 2. Table 2 summarizes the *m*-ZrO₂ surface-volume fraction and the Weibull analysis for all groups. Overall, the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia showed a higher *m*-ZrO₂ volume fraction (up to 44 vol%) compared to the Y-TZP LAVA Plus (3M ESPE) zirconia (up to 11 vol%). Moreover, the *m*-ZrO₂ in the Y-TZP LAVA Plus (3M ESPE) zirconia was not detectable after the heat treatment, whereas that of the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia remained at 14 vol% after the heat treatment at 1100 °C.

Weibull analysis revealed a higher Weibull modulus for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia than for Y-TZP LAVA Plus (3M ESPE) zirconia, independent of the surface treatment. For Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia, the highest Weibull modulus was obtained for the 'GROUND + HEAT' surface treatment, which however also corresponded to the lowest characteristic bending strength. For both zirconia grades, 'GROUND' zirconia resulted in the lowest Weibull modulus. Although the surface-treated ('GROUND', 'GROUND + Al₂O₃ SANDBLASTED' or 'GROUND + CoJet SANDBLASTED') zirconia showed a comparable characteristic strength, the 'GROUND + Al₂O₃ SANDBLASTED' and 'GROUND + CoJet SANDBLASTED' zirconia resulted in a higher Weibull modulus compared to the solely 'GROUND' zirconia. Moreover, Al₂O₃ sandblasting was more efficient in increasing the Weibull modulus than tribochemical silica (CoJet) sandblasting.

The results of the correlation analysis of the tetragonal band position around 147 cm⁻¹ in relation to the four-point bending strength are shown in Fig. 3a and b. A clear peak shift toward a higher wavelength was observed for the 'GROUND', 'GROUND + Al₂O₃ SANDBLASTED' and 'GROUND + CoJet SANDBLASTED' zirconia as compared to the 'GROUND + HEAT' zirconia. The correlation analysis revealed that a larger peak shift toward a higher wavelength, implying a higher residual compressive surface-stress level, increased the bending strength. Calculated R² values were 0.93 (*p* = 0.0338) for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia and 0.97 (*p* = 0.0155) for Y-TZP LAVA Plus (3M ESPE) zirconia.

The residual stress evolution in the top 20-µm layer of surface-treated zirconia is presented in Fig. 3c and d for the

Y-TZP LAVA Plus (3M ESPE) zirconia and the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia, respectively. 'GROUND', 'GROUND + Al₂O₃ SANDBLASTED' and 'GROUND + CoJet SANDBLASTED' Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia resulted in a gradually decreasing residual compressive stress layer of up to about 8 µm deep, whereas the compressive stress dropped much faster for the 'GROUND', 'GROUND + Al₂O₃ SANDBLASTED' and 'GROUND + CoJet SANDBLASTED' Y-TZP LAVA Plus (3M ESPE) zirconia within about 4 µm from the surface.

4. Discussion

The present study focused on the influence of different surface treatments on the four-point bending strength of dental zirconia. For both the respective Y-TZP and Y-TZP/Al₂O₃ zirconia grades (LAVA Plus, 3M ESPE; ZirTough, Kuraray Noritake), grinding with or without additional sandblasting resulted in a comparable but substantially higher characteristic bending strength in comparison with the heat-treated ground zirconia. The heat-treated ground zirconia however had the highest Weibull modulus, whereas sandblasting substantially increased the Weibull modulus of the ground zirconia. The Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia has a significantly higher Weibull modulus than the Y-TZP LAVA Plus (3M ESPE) zirconia. Therefore, the null hypothesis should be partially rejected, depending on the type of surface treatment.

In the present study, the *m*-ZrO₂ volume fraction and the residual surface stress were assessed using micro-Raman spectroscopy. Regarding the *m*-ZrO₂ surface-volume fraction, the heat treatment of the ground Y-TZP LAVA Plus (3M ESPE) zirconia at 1100 °C induced a *m*-ZrO₂ to *t*-ZrO₂ phase transformation, which resulted in an undetectable level of *m*-ZrO₂ after the heat treatment. For Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia, on the other hand, heat treatment at 1100 °C only partially induced this *m*-ZrO₂ to *t*-ZrO₂ phase transformation. This difference should probably be related to a difference in transformability of both zirconia. To confirm this assumption, we additionally assessed the transformability of Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia and Y-TZP LAVA Plus (3M ESPE) zirconia using micro-Raman spectroscopy, i.e., by measuring the amount of the *t*-ZrO₂ phase transforming into *m*-ZrO₂ upon sample fracturing. The transformability of Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia was around 60%, whereas that of Y-TZP LAVA Plus (3M ESPE) zirconia was about 10%. The higher transformability

Fig. 1 – Representative SEM photomicrographs of the four surface treatments for each zirconia grade (LAVA Plus, 3M ESPE; ZirTough, Kuraray Noritake). (a) ‘GROUND’ and (b) ‘GROUND + HEAT’ treated Y-TZP LAVA Plus (3M ESPE) zirconia: for both specimens, several scratches can be observed; (c) ‘GROUND + Al₂O₃ SANDBLASTED’ and (d) ‘GROUND + CoJet SANDBLASTED’ Y-TZP LAVA Plus (3M ESPE) zirconia: ‘GROUND + Al₂O₃ SANDBLASTED’ zirconia revealed a rougher surface than ‘GROUND + CoJet SANDBLASTED’ Y-TZP LAVA Plus (3M ESPE) zirconia. (e) ‘GROUND’ and (f) ‘GROUND + HEAT’ treated Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia: for both specimens, dark contrast alumina particles can be observed; (g) ‘GROUND + Al₂O₃ SANDBLASTED’ and (h) ‘GROUND + CoJet SANDBLASTED’ Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia: the surface characteristics were similar to those observed for the ‘GROUND + Al₂O₃ SANDBLASTED’ and ‘GROUND + CoJet SANDBLASTED’ Y-TZP LAVA Plus (3M ESPE) zirconia.

of Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia resulted in a larger amount of *m*-ZrO₂ after the surface treatment, as compared to that for Y-TZP LAVA Plus (3M ESPE) zirconia. Apparently, a temperature of 1100 °C is not enough to completely transform the surface treatment induced *m*-ZrO₂ for the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia. We additionally heat-treated the ground Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia at 1450 °C and still could not detect *m*-ZrO₂. The surface-phase composition of the ground

and heat-treated (at 1450 °C) Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia was the same as that of the as sintered (untreated) Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia.

Stress analysis of surface-treated zirconia can be performed by X-ray diffraction [10,23] or micro-Raman spectroscopy [24]. According to Pezzotti and Porporati, micro-Raman peaks shift linearly with the magnitude of the residual stress level [19]. The peak shift of the tetragonal band around 147 cm⁻¹ was assessed because this tetragonal peak does

Fig. 2 – (a) Graph showing the Weibull analysis for the different surface-treated Y-TZP LAVA Plus (3M ESPE) zirconia. Higher scale parameters were recorded for the ‘GROUND’, ‘GROUND + Al₂O₃ SANDBLASTED’ and ‘GROUND + CoJet SANDBLASTED’ zirconia than for the ‘GROUND + HEAT’ treated zirconia. On the other hand, a higher shape parameter was recorded for the ‘GROUND + HEAT’ treated zirconia than for the other surface-treated zirconia. (b) Graph showing the Weibull analysis for the different surface-treated Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia. The trends were similar as to those for Y-TZP LAVA Plus (3M ESPE) zirconia. Overall, higher shape parameters were obtained for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia than for Y-TZP LAVA Plus (3M ESPE) zirconia.

Table 2 – Summary of the *m*-ZrO₂ volume fraction and the four-point bending strength Weibull analysis results.

Zirconia	Surface treatment	n	<i>m</i> -ZrO ₂ volume fraction (%)	Weibull modulus <i>m</i> ¹	Characteristic strength σ_0 ² (B63.2)	95% confidence level at σ_0 ³ (B63.2)
Y-TZP LAVA Plus (3M ESPE) zirconia	‘GROUND’	10	3.5	6.2	1192	1061–1348 (ab)
	‘GROUND + HEAT’	9	0	9.4	934	862–1017 (c)
	‘GROUND + Al ₂ O ₃ SANDBLASTED’	9	10.9	13.0	1107	1042–1178 (b)
	‘GROUND + CoJet SANDBLASTED’	10	6.9	7.1	1149	1041–1276 (ab)
Y-TZP/Al ₂ O ₃ ZirTough (Kuraray Noritake) zirconia	‘GROUND’	10	37.2	11.3	1237	1174–1304 (ab)
	‘GROUND + HEAT’	10	13.9	24.7	994	966–1026 (c)
	‘GROUND + Al ₂ O ₃ SANDBLASTED’	10	36.5	19.9	1262	1218–1311 (a)
	‘GROUND + CoJet SANDBLASTED’	10	35.6	15.3	1212	1156–1274 (ab)

¹ the higher the Weibull shape, the more reliable the treatment.

² the higher the Weibull scale, the higher the bonding effectiveness.

³ same letters within brackets indicate absence of statistical significance at B63.2.

not overlap with any *m*-ZrO₂ bands [25]. A peak shift of this tetragonal band toward a higher wavelength corresponds to the presence of a residual compressive stress [19]. For both zirconia grades, micro-Raman analysis showed a clear peak shift toward a higher wavelength for the *t*-ZrO₂ phase for the three surface-treated (‘GROUND’, ‘GROUND + Al₂O₃ SANDBLASTED’ and ‘GROUND + CoJet SANDBLASTED’) zirconia. ‘Ground + HEAT’ treated zirconia showed a similar peak position as for the as sintered (untreated) zirconia, which can be assumed to be the ‘stress-free’ condition. The correlation between the *t*-ZrO₂ peak position due to the residual compressive stress level and the characteristic four-point bending strength was clear and significant for both zirconia grades ($R^2 = 0.97$, $p = 0.0155$ for Y-TZP LAVA Plus (3M ESPE) zirconia, $R^2 = 0.933$, $p = 0.0338$ for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia), as presented in Fig. 3a and b. Wulfman et al. assessed the sampling depth of micro-Raman spectroscopy.

According to them, probes based on 10× objective penetrated up to 68 μm, whereas those based on the 80× objective were limited to 5 μm [26]. Their results indicate that in our setting (100× objective), the sampling depth of micro-Raman spectroscopy would be less than 5 μm.

Moreover, the depth of the residual compressive stress was assessed using confocal micro-Raman spectroscopy, as shown in Fig. 3c and d. For Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia, grinding, Al₂O₃- and CoJet-sandblasting created a deeper residual compressive stress zone (up to 8 μm deep) than for the Y-TZP LAVA Plus (3M ESPE) zirconia (up to about 4 μm deep). The thicker compressive stress layer at the surface of the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia can be directly related to the higher Weibull modulus, as compared to that at the surface of the Y-TZP LAVA Plus (3M ESPE) zirconia.

Fig. 3 – (a) Summary of correlation analysis for Y-TZP LAVA Plus (3M ESPE) zirconia. The average of the detected distinct peak shift of the tetragonal ZrO₂ band around 147 cm⁻¹ was measured after surface treatment. Correlation analysis revealed that a larger peak shift, i.e., a higher compressive stress in the surface layer, resulted in higher four-point bending strength ($R^2 = 0.97$, $p = 0.0155$). **(b) Summary of correlation analysis for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia.** The average of the detected distinct peak shift of the tetragonal ZrO₂ band around 147 cm⁻¹ was measured after surface treatment. Correlation analysis revealed that a larger peak shift, i.e., a higher compressive stress in the surface layer, resulted in higher four-point bending strength ($R^2 = 0.93$, $p = 0.0338$). **(c) and (d) Evolution of the t-ZrO₂ peak position as a function of depth from the surface.** ‘GROUND’, ‘Al₂O₃ SANDBLASTED’ or ‘CoJet SANDBLASTED’ Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia resulted in a deeper residual stress zone (up to 8 μm) as compared to ‘GROUND’, ‘Al₂O₃ SANDBLASTED’ or ‘CoJet SANDBLASTED’ Y-TZP LAVA Plus (3M ESPE) zirconia (up to 4 μm).

Literature on actual residual stress measurement of surface-treated zirconia using micro-Raman spectroscopy is scarce [24]. A higher residual compressive surface-stress level was measured on a surface-treated (SiC-sandblasted) Ce-TZP/Al₂O₃ zirconia compared to a Y-TZP zirconia, this most probably due to the higher transformability of Ce-TZP/Al₂O₃ zirconia, whereas a significantly lower residual stress was measured for heat-treated Y-TZP and Ce-TZP/Al₂O₃ zirconia [24]. The latter is in line with our present results. Heat treatment caused *m*-ZrO₂ to *t*-ZrO₂ phase transformation and compressive stress release, resulting in a concomitant decreased flexural strength of zirconia [3].

The four-point bending strength data were analyzed using Weibull statistics. The Weibull distribution is a continuous probability distribution; Weibull survival analysis has been developed to determine the level of structural reliability of materials [27–29]. Several studies have indeed applied Weibull analysis to assess the reliability of the mechanical

properties of dental zirconia [10–12,16,30]. The main difference with other statistical approaches is that the distribution of the failures is not determined before, but is estimated from the data. The main advantage is that not only information regarding bending strength is gathered, but also the reliability of the outcome is estimated. Each Weibull analysis is characterized by two principal parameters, the Weibull “shape” or modulus (*m*), and the Weibull “scale”, or the B63.2 parameter or characteristic strength (σ_0).

Applied to this work, the higher the Weibull modulus *m*, the more reliable the zirconia in terms of mechanical properties; the higher the characteristic strength σ_0 , the higher the actual bending strength. Although the σ_0 was similar for both zirconia grades, Weibull moduli were clearly different. The Weibull modulus as a function of the applied surface treatment of the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia ranged from 11.3 to 24.7, whereas that of the Y-TZP LAVA Plus (3M ESPE) zirconia varied from 6.2 to 13.0 (see Table 2).

The three surface-treated zirconia ('GROUND', 'GROUND + Al₂O₃ SANDBLASTED' and 'GROUND + CoJet SANDBLASTED') resulted in a comparable high characteristic strength, which was substantially higher than that for the 'GROUND + HEAT' treated zirconia. The Weibull modulus of the 'GROUND' zirconia however was the lowest. These two trends are in line with previous studies, which assessed the mechanical properties of ground or Al₂O₃-sandblasted dental zirconia with/without additional heat treatment using a biaxial flexural strength test [9,12].

Additional Al₂O₃-sandblasting or tribochemical silica (CoJet) sandblasting after grinding resulted in a higher Weibull modulus compared to that for the 'GROUND' zirconia. This is most probably due, at least in part, to the smoothening effect of the additional sandblasting, which removes grinding marks and micro-cracks that may otherwise act as stress concentrators [31]. In addition, this could also in part be attributed to the deeper and possibly more continuous layer that is subjected to a compressive stress state after sandblasting, as was revealed by micro-Raman analysis.

The tribochemical silica (CoJet) sandblasting is more gentle, as compared to Al₂O₃-sandblasting, which resulted in a limited increase in Weibull modulus (Fig. 1). The size of the surface flaws, which were introduced by sandblasting, did not seem to have exceeded the thickness of the compressive surface layer, since the strength of the material was not reduced [12]. Moreover, Chintapalli et al. reported that sandblasting using a smaller particle size (110 µm) did not produce additional micro-cracks at the zirconia surface [32]. Grinding, on the other hand, was shown to produce micro-cracks at the zirconia surface [33].

Although the nature of the flaws in the 'GROUND' zirconia and 'GROUND + HEAT' zirconia should be the same, the 'GROUND + HEAT' zirconia grades had a significantly lower characteristic strength, but higher Weibull modulus. This is most probably due to the residual compressive stress zone at the surface of the ground zirconia [33]. In the present study, ZirTough (Kuraray Noritake) is an Y-TZP/Al₂O₃ zirconia that contains ≤10 wt% Al₂O₃ as a secondary phase. The addition of Al₂O₃ to Y-TZP zirconia induces a smaller grain size, as compared to that of Y-TZP zirconia, which results in a smaller flaw size as compared to Y-TZP zirconia [34]. This effect is more obvious when Y-TZP-Al₂O₃ zirconia receives an additional HIP (hot isostatic pressing) treatment, as the HIP treatment also reduces the porosity of Y-TZP/Al₂O₃ zirconia [35]. In the present study, however, the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia was not HIP-ed, but the size of the Al₂O₃ grains was relatively large (around 500 nm), having resulted in a comparable characteristic bending strength level as for the Y-TZP LAVA Plus (3M ESPE) zirconia. Nevertheless, the additional Al₂O₃ phase resulted in a higher reliability in bending strength, as compared to that of the Y-TZP zirconia.

5. Conclusion

Surface grinding with or without additional Al₂O₃ or tribochemical silica (CoJet) sandblasting induced residual compressive stress at the zirconia surface, and resulted in a comparable characteristic four-point bending strength of the

Y-TZP LAVA Plus (3M ESPE) zirconia and the Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia. Surface grinding with or without additional Al₂O₃ or tribochemical silica (CoJet) sandblasting for Y-TZP/Al₂O₃ ZirTough (Kuraray Noritake) zirconia resulted in a deeper compressive stress zone than for Y-TZP LAVA Plus (3M ESPE) zirconia, that on its turn resulted in an increased Weibull modulus and concomitantly increased zirconia reliability in terms of mechanical properties. Thermal treatment after grinding however eliminated the presence of the residual compressive stress surface layer, and resulted in a decrease of the characteristic strength by approximately 200 MPa.

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