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Fatigue behavior in water of Y-TZP zirconia ceramics after abrasion with 30 μ m silica-coated alumina particles

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Abstract

Objective—The use of a 30 μ m alumina–silica coated particle sand (CoJet™ Sand, 3M Espe), has shown to enhance the adhesion of resin cements to Y-TZP. The question is whether or not sandblasting 30 μ m particles does negatively affect the fatigue limit (*S–N* curves) and the cumulative survival of Y-TZP ceramics.

Method—Four zirconia materials tested were: Zeno (ZW) (Wieland), Everest ZS (KV) (KaVo), Lava white (LV) and Lava colored (LVB) (3M Espe). Fatigue testing (*S–N*) was performed on 66 bar of 3 mm $\times$ 5 mm $\times$ 40 mm with beveled edges for each zirconia material provided by the manufacturers. One half of the specimens were CoJet sandblasted in the middle of the tensile side on a surface of 5 mm $\times$ 6 mm. Cyclic fatigue (*N* = 30/group) (sinusoidal loading/unloading at 10 Hz between 10% and 100% load) was performed in 3-point-bending in a water tank. Stress levels were lowered from the initial static value (average of *N* = 3) until surviving 1 million cycles. Fatigue limits were determined from trend lines. Kaplan–Meier survival analysis was performed to determine the failure stress at the median percentile survival level for 1 million of cycles before and after sandblasting. The statistical analyses used the log-rank test. Characterization of the critical flaw was performed by SEM for the majority of the failed specimens.

Results—The fatigue limits “as received” (ctr) were: LV = 720 MPa, LVB = 600 MPa, KV = 560 MPa, ZW = 470 MPa. The fatigue limits “after CoJet sandblasting” were: LV = 840 MPa, LVB = 788 MPa, KV = 645 MPa, ZW = 540 MPa. The increase in fatigue limit after sandblasting was 15% for Zeno (ZW) and Everest (KV), 17% for Lava (LV) and 31% for Lava colored (LVB). The KM median survival stresses in MPa were: ZW(ctr) = 549 (543–555), ZW(s) = 587 (545–629), KV(ctr) = 593 (579–607), KV(s) = 676 (655–697), LVB(ctr) = 635 (578–692), LVB(s) = 809 (787–831), LV(ctr) = 743 (729–757), LV(s) = 908 (840–976). Log-rank tests were significantly different (*p* < 0.001) for all sandblasted groups vs. the “as received” except for Zeno (Wieland) (*p* = 0.295). Failures started from both intrinsic and machined flaws.

Significance—30 μ m particle sandblasting did significantly improve the fatigue behavior of three out of four Y-TZP ceramic materials and can therefore be recommended for adhesive cementation procedures. This study was supported in part by grants from the Swiss Society for Reconstructive Dentistry (SSRD) and 3M Espe.

Keywords

Fatigue testing; Dental; Ceramics; Zirconia

1. Introduction

Due to its excellent mechanical properties (i.e. bending strength and toughness) Yttria stabilized tetragonal zirconia polycrystal (Y-TZP) has become an attractive high-toughness core material for fixed dental restorations [1–6]. The innovation of zirconia ceramics lies in its stress-induced toughening mechanism, which impedes crack propagation [7–9]. During stress application, the tetragonal crystallites (*t*) transform into their monoclinic (*m*) form at the crack tip. The concomitant 3–5% expansion in volume [10] leads to the development of internal compressive stresses that oppose crack propagation and thus increase the material's fracture toughness (K_{IC}) and strength. Transformation can be induced by any externally applied stress such as generated by grinding, upon impact or during outright fracture. Grinding Y-TZP and sandblasting will create compressive stresses in the surface due to the $t \rightarrow m$ phase transformation [11,12]. The magnitude of the increase in strength depends on the volume % of transformed ZrO_2 and the depth of the compressive layer underneath the surface (i.e. the "transformed zone depth") [11,12]. Still, forceful grinding will introduce deep surface flaws, which extend beyond the surface compressive layer. These will act as stress concentrators and curtail the strength of the workpiece [11,13–15].

Sandblasting of zirconia ceramic with 50 or 110 μm alumina particles improves adhesion to resin-based cements due to increased surface roughness [16,17]. Also, high adhesive bond strengths were demonstrated when sandblasting with 30 μm silica-coated alumina particles at ca. 2.8 bar in combination with phosphate monomer resin-based cements [18–22]. Concerns have been raised regarding the surface damages after sandblasting with alumina particles of 50–120 μm at pressures of 2.8–4 bar [13,23–25]. Kosmač [13] reported a decline in the reliability (from Weibull $m = 11$ –7.5) of zirconia specimens after sandblasting with 110 μm alumina particles. The deterioration was in direct relation with the damages affecting the ceramic surface. The authors warned that these impact flaws might develop into severe stress intensifiers when exposed to a wet environment under cyclic loading and thus facilitate crack nucleation, even at lower levels of applied stress. Along the same lines, Zhang et al. [23,25] reported a 30% decrease in strength during contact fatigue testing after sandblasting mirror polished surfaces of Y-TZP with 50 μm alumina particles at 2.8 bar. The decline was explained by the formation of microcracks ($>4 \mu m$) in the specimens which under sustained loading would grow and promote radial crack initiation [24].

Clinically, the typical surfaces of zirconia frameworks for dental restoration are not mirror polished but utilized as machined by the CAD–CAM devices. They thus harbor a variety of initial surface flaws, which may be further affected by sandblasting. On CAD–CAM machined Lava™ zirconia specimens with $R_a = 0.21 \mu m$, Curtis et al. [26] found no difference in biaxial strength and reliability when tested in water after sandblasting with 25, 50 or 110 μm Al_2O_3 . In contrast Wang et al. [27] demonstrated an increase in dry static flexural strength and reliability after sandblasting a CAD–CAM machined Y-TZP (Cercon®) ($R_a = 1.9 \mu m$) ceramic with 50 μm alumina particles. These seemingly contradictory findings may actually be related to a variety of factors such as the nature of the defects induced by machining and processing, the t – m phase distribution in the surface and the residual stress state prior to testing.

Concerns have been also expressed regarding the degradation of zirconia ceramic in the oral environment due to the exposure to water and mechanical stress over prolonged periods [28–

32]. Still, Y-TZP is currently the strongest ceramic in clinical use [33–35]. Further, its sandblasting with 30 μm silica-coated alumina particles has been recommended to improve the adhesion to resin-based cements [18,20–22]. It is unclear whether or not a “less aggressive” sandblasting with 30 μm silica-coated alumina particles does affect the fatigue resistance of CAD–CAM machined Y-TZP ceramics. In this context, stress vs. number of cycles diagrams (S – N - or Wöhler curves) will allow the experimentalist to define a stress range (i.e. the “fatigue limit”) below which failure will not occur on any realistic time scale.

The objectives of the present study therefore were: (1) To draw S – N diagrams and determine the fatigue limits of four Y-TZP zirconia materials “as received” and “sandblasted” with 30 μm silica-coated alumina particles. Further to determine median Kaplan–Meier survival stresses under cyclic fatigue loading. (2) To characterize the type of flaws affecting fatigue failure using fractographic analysis. (3) To determine the proportions of tetragonal vs. monoclinic phases “as received” and “sandblasted” using X-ray diffraction.

2. Materials and methods

The zirconia materials used in this study are summarized in Table 1. Bars 40 mm $\times$ 3 mm $\times$ 5 mm with beveled edges were provided “as received” by the manufacturers for each Y-TZP material. Prior to testing, each bar was subjected to four firing cycles (930 $^{\circ}\text{C}$, 900 $^{\circ}\text{C}$, 890 $^{\circ}\text{C}$, 880 $^{\circ}\text{C}$) corresponding to the firing programs for “liner”, “dentin 1”, “dentin 2” and “glaze” of a zirconia veneering ceramic (Zirox[®], Wieland) to duplicate the effect of veneering ceramic application.

2.1. Material characterization

2.1.1. Microstructure, density, surface roughness (R_a)—The microstructure of each material was determined after thermal etching mirror polished surfaces. Thermal etching was performed 50 $^{\circ}\text{C}$ below the sintering temperature for 30 min. Scanning Electron Microscope images were taken at 20,000 $\times$ magnification and the average grain size was determined using the linear intercept count method (ASTM E112-96(2004)e2).

The density of the sintered zirconia specimens was measured on $N = 5$ specimens per material (3 measurements per specimen) using Archimedes’ method.

The surface roughness ($N = 3/\text{material}$) was measured on the “as received” bars using an electro-mechanical profilometer (M1, Mahr) equipped with a drive unit (PGK, Mahr) and a measuring sensor (MFW-250, Mahr) with a 40 nm z resolution. The bars were mounted on an XY cross table. As received and after sandblasting, areas of 5.6 mm $\times$ 1.75 mm oriented parallel to the long axis of the bars were scanned using a 2 μm radius diamond stylus at a speed of 0.5 mm/s and an applied force of 0.5 mN. Frames of 51 line profiles with y -axis steps of 60 μm were obtained. To determine the average surface roughness (R_a), a Gaussian profile filter (ISO 11562) with the cut-off wavelength set at $\lambda_c = 0.8$ mm and an evaluation length of $l_n = 5.6$ mm was used.

2.1.2. Weibull parameters—Weibull’s characteristic (σ_0) strengths and moduli (m) were determined from 10 mirror polished (1 μm) bars of each material tested. To this effect, a customized 3-point bending device was mounted into a water tank (Fig. 1). The lateral axes (rollers) supporting the specimens were incorporated in anti-torsion devices allowing rotation, thus compensating for minor unevennesses in the surfaces. The span between the rollers was set to 30 mm and the crosshead speed was 0.5 mm/min.

2.2. S–N diagrams, fatigue limits, survival analysis

Sixty-six CAD–CAM bars as machined by the manufacturers were used for fatigue testing. Half of the specimens were sand-blasted in their midportion (i.e. at the maximum tensile stress) with 30 μm silica-coated alumina particles (CoJet™ Sand, 3M ESPE) on a surface of 6 mm $\times$ 5 mm for 20 s. Sandblasting was performed using an air abrasion gun (RondoFlex 2013, KaVo) at 2.5 bar with the tip inclined approximately 45° and at a distance of 7 mm. The specimens were then ultrasonically cleaned in pure ethanol for 10 min using a sonic device (Micro 10+, Unident) set to a low frequency range (28–34 kHz). Fatigue tests were conducted on a servo-hydraulic machine (Schenck PSB1017) using the 3-point bending assembly immersed in a water tank (Fig. 1). The span between the rollers was set to 30 mm. The fatigue test conditions were as follows: (i) sinusoidal loading/unloading cycles at a frequency of 10 Hz, (ii) a stress ratio of $R = \sigma_{\min}/\sigma_{\max} = 0.1$ (amplitude of 90%) and (iii) at the onset of the test, a 5 s ramp (i.e. 50 cycles) before the maximum imposed stress level ($\sigma_{\max}$) was reached.

Prior to fatigue testing, the monotonic failure stresses were determined from 3 specimens of each material and surface conditions (i.e. “as received” vs. “sandblasted”). Testing was performed in water at a crosshead speed of 0.5 mm/min. From the average monotonic stresses recorded, the entry fatigue stress level was set 20% below the monotonic stress at failure. S–N diagrams (stress vs. number of cycles to failure) were generated from 30 specimens per material and per surface condition. Fatigue limits were set at one million cycles and determined from trend lines using a least square linear regression fit. The test was censored at 10^6 cycles and Kaplan–Meier survival analysis was performed to determine the failure stress at the median percentile. Kaplan–Meier analysis is a statistical method used to estimate the cumulative probability of survival for specimens or prostheses at each point in time, which is calculated as the product of conditional probability of survival for all previous time intervals [36]. Kaplan–Meier analysis is useful in preventing probability of survival estimates from being biased by specimens or prostheses that survive beyond the window of observation [37], also known as right-censored data or run-out specimens.

2.3. Failure origins

The fracture surface of the first 20 failed specimens in each group was first examined under a stereo microscope at maximum 57 $\times$ magnification and then investigated by scanning electron microscopy to determine the origin of the failure (i.e. intrinsic flaws due to processing or extrinsic flaws due to machining). Fractographic analysis was performed in accordance with the NIST *Recommended practice guide for fractography of glasses and ceramics* [38]. Features such as mirrors and hackle were identified on the fracture surface and aided in locating the origin of the failure.

2.4. X-ray diffraction

X-ray diffraction (XRD) was employed to quantify the crystalline phases on the “as received” and after sandblasting each Y-TZP material. XRD analyses were conducted using a diffractometer (Siemens D5005, Bruker AXS) with Ni filtered Cu K α radiation (1.5418 Å) at 35mA and 40 kV. Diffraction data were collected from 26° to 65° 2 θ , with a step size of 0.010° and a scanning time of 2 s/step. A quantitative value of phase compositions of the “as received” and “after sandblasting” Y-TZP ceramics was obtained after applying Rietveld refinement analysis (Topas, Bruker AXS).

3. Results

The materials characteristics regarding microstructure, density, roughness and Weibull parameters are summarized below.

3.1. Microstructure, density and roughness (Ra)

Fig. 2 displays the microstructure of the four Y-TZP materials at 20,000 $\times$ magnification. The average grain size, standard deviation and range as determined by the “Linear interceptive count method” were as follows: Everest (KV) 383 nm (± 47) (140–800 nm), Zeno (ZW) 383 nm (± 47) (160–880 nm); Lava (LV) 537 nm (± 47) (160–1200 nm); Lava colored (LVB) 643 nm (± 61) (200–1600 nm).

Densities and roughness values are listed in Table 2. The roughness of Everest (KV) “as received” specimens was the highest of the four materials because the machining grooves of these specimens were oriented in transverse direction while the other materials were ground longitudinally. With the exception of Everest (KV), the roughness slightly but significantly increased (Table 2) after sandblasting with CoJet.

3.2. Weibull parameters

Weibull’s characteristic strength (σ_0), moduli (m) and corresponding confidence intervals (CI) are summarized in Table 3 for the mirror polished bars. The characteristic strength of the Zeno ceramic was significantly inferior to the three others. No significant differences were evidenced in the moduli.

3.3. S–N diagrams, fatigue limits, survival analysis

Fatigue data for the “as received” and “CoJet sandblasted” zirconia are presented as S – N diagrams in Fig. 3a and b. Initial monotonic failure stresses (at $N = 1$) are connected with the cyclic fatigue data ($N = 30$). The fatigue limits set at 1 million cycles obtained in 3 point-bending at 10 Hz in water were determined from trend lines using a least square linear regression fit. The fatigue limits of “as received” samples (Fig. 3a) were: LV = 720 MPa, LVB = 600 MPa, KV = 560 MPa, ZW = 470 MPa. The fatigue limits of “CoJet sandblasted” specimens (Fig. 3b) were: LV = 840 MPa, LVB = 788 MPa, KV = 645 MPa, ZW = 540 MPa. The increase in fatigue limit of the sandblasted specimens was 15% for Zeno ZW (Wieland) and Everest KV (KaVo), 17% for Lava LV (3M Espe) and 31% for Lava colored LVB (3M Espe).

Kaplan–Meier cumulative survival plots for all four materials for 1 million cycle data “as received” and “CoJet sandblasted” are displayed in Fig. 4. The beneficial effect of CoJet sandblasting is clearly shown for all four materials with a statistical significance at 95% CI (Holm–Sidak) for all zirconia materials except Zeno (ZW) (Wieland) ($p = 0.295$).

The survival stresses of the Kaplan–Meier median percentiles (MPa $\pm 95\%$ CI) as a function of fatigue cycles and surface treatment are listed in Table 4. Two statistical analyses were performed on the data. The log-rank test yield a $p < 0.001$ overall between survival curves. Subsequent pairwise testing with Holm–Sidak tests demonstrated a significant effect of sandblasting ($p < 0.05$) for all materials except ZW ($p = 0.295$).

When plotting both, the S – N diagram and KM survival curves of the best fatigue performing zirconia material (Lava LV), “as received” and “CoJet sandblasted” (Fig. 5) the augmentation in fatigue limit approaches 17%. The Kaplan–Meier survival plot from the same material at 1 million cycles confirms the significant improvement of the CoJet sandblasting treatment on its survival probability with amedian percentile “as received” and “CoJet sandblasted” of respectively 743 MPa and 908 MPa.

3.4. Failure analysis

The first 20 or more of the failed specimens were analyzed fractographically for determination of the failure origin and flaw type using a stereomicroscope as well as a

scanning electron microscope (SEM) for higher magnifications. Fig. 6 displays the two fractured halves of a Lava (3M Espe) zirconia specimen that failed under fatigue loading. The symmetric mirrors (i.e. the smooth surfaces) and the radiating hackle lines are clearly visible on the tensile aspects of the samples. The mirror, which is easy to see in the stereomicroscope surrounds the origin of failure, for which higher SEM magnifications are needed.

SEM observation demonstrated processing flaws (associated with fabrication and sintering) as well grinding defects (Table 5). For Y-TZP Zeno (ZW) half of the specimens analyzed after fatigue failure exhibited flaws due to cold pressing of the blanks and which acted as stress concentrators during crack nucleation (Fig. 7). The critical flaw sizes leading to failure as seen in Fig. 7A–D were: $\sim 38\mu\text{m} \times 27\mu\text{m}$, $32\mu\text{m} \times 28\mu\text{m}$, $21\mu\text{m} \times 18\mu\text{m}$ and $15\mu\text{m} \times 15\mu\text{m}$ respectively. For each specimen, more detailed information regarding numbers of cycles and stresses at failure is provided in the figure legend.

In the remaining three materials (LV, LVB and KV) only a few inherent flaws such as pores, spray dry agglomerates and large grains from excessive grain growth were found at the failure origins (Fig. 8A–D). Critical flaw sizes, number of cycles- and stresses at failure are mentioned in the legends. The small ($10\mu\text{m} \times 15\mu\text{m}$) subsurface sintering agglomerate (Fig. 8A) had no effect during fatigue testing as the specimen survived 10^6 cycles at 951 MPa. In this instance, the specimen was monotonically fractured after surviving the fatigue test and the failure stress reached 1489 MPa.

Table 5 presents the type of flaws observed in all four Y-TZPs in the “as received” specimens as examined by SEM. The majority of failure flaws for ZW resulted from blank fabrication. In the other three zirconia materials, the grinding flaws prevailed.

Fig. 9 illustrates the flaws types as distributed within the *S–N* curve for the Zeno (ZW) Y-TZP material. Below 2000 cycles, the blank pressing (processing) flaws were dominant. After 2000 cycles, the grinding flaws prevailed. Surface grinding flaws acting as stress concentrators resulted from the specimens’ machining (Fig. 10). The typical grinding flaws were rather shallow (between 7 and $20\mu\text{m}$ deep) and elongated ($>100\mu\text{m}$) along the surface. Several specimens in Fig. 10A, B and D demonstrated stable crack extension prior to fracture, therefore one has to be cautious when determining the toughness based on failure stress and crack size in a wet environment. Depending on the crack history and environment, the crack length at fracture (that is the critical crack size) may be different from the initial flaw (such as a grinding flaw) from which failure started [39]. Nonetheless, providing that a correct critical crack size is measured, the classic K_{Ic} equation ($K_{Ic} = Y\sigma\sqrt{a}$) can be applied to fatigued specimens with slow crack growth for toughness approximations.

An overview of the surfaces of the four zirconia materials as provided by the manufacturers is shown in Fig. 11. Zeno specimens displayed a blend of longitudinal, transverse and oblique machining grooves (Fig. 11A). Everest (Fig. 11B) was reshaped from the sides leaving a central portion with transverse (parallel) grooves combined with a smoother surface made of oblique and longitudinal grinding marks. This combination of two types of grinding is expressed by the higher *Ra* value and standard deviations (Table 2). Lava (LV) (Fig. 11C) and Lava colored (LVB) (Fig. 11D) (3M Espe) were similar in their grinding marks, which were essentially oriented longitudinally. Still, for both materials, some occasional deeper grooves were also observed. Nevertheless, one has to keep in mind that the roughness measurements in the present study were performed only on the longitudinal bar axis for an evaluation length of 5.6 mm in accordance with ISO standard 11562. No measurements were performed on the specimens’ minor axis, as the evaluation length would not have been applicable due to the specimen’s small width (5 mm). Therefore, the reported

Ra values in this study may not reflect properly the multiple direction machining grooves present on the different zirconia materials tested. Ideally speaking one should provide Ra values for both major and minor axis of bend bars.

3.5. X-ray diffraction

The Rietveld analysis (Table 6) conducted on the data generated by X-ray diffraction of the “as received” samples demonstrated the presence of two phases within the ceramics. The primary phase was tetragonal, identified by the lattice parameters a and c . The secondary phase was cubic. The mass fraction of each phase is indicated in wt.%.

Typical X-ray diffraction profiles “as received” and after “CoJet sandblasting” are shown in Fig. 12. The sandblasted profile (top) displayed an asymmetrical broadening at the (1 0 1), (2 0 2) and (1 1 3) peaks and the reversal of intensities of peaks (0 0 2) and (2 0 0). The broadening could indicate the emergence of a new phase, which is cubic 2. In addition, a slight $t \rightarrow m$ transformation was noted. A small diffraction peak appeared at $\sim 28 2\theta$, which most likely was attributable to the crystallo-graphic plane $(-1\ 1\ 1)$ of the monoclinic phase. However, the quantitative recordings of the monoclinic phase were below 5% and could therefore not be properly fitted with the Rietveld procedure.

4. Discussion

The essential issue of this project was to determine whether the fatigue limit of CAD–CAM machined Y-TZP ceramics was negatively affected by sandblasting with 30 μm silica-coated alumina particles. The results clearly demonstrate that this did not occur. On the contrary, the “soft” (30 μm at 2.5 bar) sandblasting procedure augmented the fatigue limit by 15–31%. Further, for all 4 Y-TZPs, the failure stress at the median probability of survival was also raised. The fractographic analysis provided information regarding the type of flaws acting as stress-raisers. From Table 5 and Fig. 10, the fatigue strength of three out of four “as received” specimens was primarily controlled by the catastrophic propagation of machining flaws. Critical blank pressing flaws (i.e. powder compacts) were primarily identified in the Zeno specimens but were also occasionally found in the other three Y-TZPs (i.e. Everest, Lava and Lava colored). The type of blank pressing procedures applied by each manufacturer is proprietary information. Still, uniaxial pressing would be less effective than biaxial or isostatic 3D pressing. Wieland (i.e. Zeno’s manufacturer) uses cold isostatic pressing and critical blank pressing defects were often encountered within the specimens (i.e. Fig. 7). These cracklike voids most certainly result from different shrinkage rates of the powder agglomerates relative to the surrounding compact [40,41]. If the agglomerates are produced by spray drying, they may not uniformly deform to fill interagglomerate void space during consolidation steps [40]. Additional causes for such processing defects should be sought in the rubber molds used for pressing, the granulate binders, the embedding material (when present) as well as in contaminations from inorganic inclusions, all of which are capable of generating volume- or surface flaws [42]. The Zeno zirconia indeed included many compact processing defects, that is, heterogeneities acting as stress concentrators, which decreased the fatigue limits and augmented the scattering in the early failures (below 2000 cycles).

Overall, with the exception of Zeno Wieland, the majority of the specimens failed from machining flaws. It is common knowledge that ceramic flexure specimens are highly sensitive to surface flaws. Those in turn are dependent on the grit of the grinding wheel and the grinding direction [15,43,44]. Microcracks will form on the ceramic surface or subsurface during the abrasion with diamond tools; the larger the grit, the deeper the cracks and the lower the strength. Although the roughness measurements in this study yield rather smooth zirconia surfaces, machining flaws that limited the strength (i.e. parallel cracks from

machining perpendicular to the long axis of the bend bar or orthogonal cracks from longitudinal grinding (i.e. parallel to the long axis) were identified at the majority of the failure origins. Despite the efforts made by the manufacturers to provide longitudinally ground specimens for this study, microscope views showed a variety of machining directions which created microcracks parallel-, oblique- and orthogonal to the long axis of the specimens. These multidirectional grinding marks are very typical for CAD–CAM zirconia frameworks as documented in Fig. 13 representing a clinical 3Y-TZP Lava bridge framework viewed under stereomicroscope as well as under SEM.

The overall shape of the S – N response was almost flat with differences of approximately 50MPa only between the low cycle (100–1000 cycles) and high cycle (10^6) domain. Machining flaws may be decreased in depth by using a small grain diamond abrasive to finish the surface. Although the fabrication of bend bars is certainly more challenging than a smaller coping for a crown or bridgework, the issue here is to point out the weakest link in the material will come from processing flaws, either intrinsic blank pressing, sintering, or machining [43,45]. If the processing and grinding flaws can be kept to a minimum, one can expect to improve the fatigue limits.

Overall, the lifetime (numbers of cycles to failure) at a given stress level for the “as received” and “CoJet sandblasted” of the two Lava (3M Espe) zirconia materials, were always longer compared to the Y-TZPs from KaVo and Wieland. This can be explained by a lower density of intrinsic fabrication flaws for Lava and that failure resulted mainly from longitudinal microcracks due to machining (orthogonal flaws) which are less critical than parallel machining cracks activated by transverse grinding [43,44]. The specimens were immersed in water to more closely duplicate intraoral conditions. It is indeed being increasingly recognized that zirconia suffers from low temperature degradation in a humid environment *via* alterations in its crystalline structure [29]. On a number of broken specimens of all fatigue groups, stable crack growth from pre-existing flaws could be detected. These phenomena might have been nurtured by the aqueous testing conditions. Fatigue studies of zirconia in water have shown dental Y-TZP to have an increased susceptibility to crack propagation [33,35]. These materials though can still be recommended for clinical use as they demonstrate superior lifetimes relative to alumina or glass-ceramics [33–35].

Mechanical properties of zirconia also depend on the phase stability and grain size. One may wonder about the presence of a cubic 1 phase (Table 6) in all the “as received” 3Y-TZP materials ranging from 8 to 28 wt.%. The presence of cubic-phase regions without grain-growth has been shown in 3Y-TZP by several authors [46–48]. Matsui explained the presence of a cubic phase by the “*grain-boundary segregation-induced phase transformation mechanism*” in which the cubic-phase regions start to form by a $t \rightarrow c$ phase transformation from grain boundaries and/or triple junctions in which Y^{3+} ions segregate, followed by cubic-phase regions formation in grain interiors adjacent to the grain boundaries [47,48].

The improved fatigue strength after sandblasting may be related to the presence of compressive stresses on the tensile surface. These could result from changes in the crystalline structure of the Y-TZP ceramics as indicated by the X-ray diffraction profiles. In the present study, Rietveld-refined X-ray diffraction data confirmed the presence of tetragonal and cubic phases in all ‘as machined’ specimens. After blasting with CoJet™ Sand, an asymmetrical broadening at the (1 0 1), (2 0 2) and (1 1 3) peaks and the reversal of intensities of peaks (0 0 2) and (2 0 0) were observed. The broadening could indicate the emergence of a new ‘cubic 2’ phase. Induced stresses resulting from grit blasting are likely to locally distort the lattice and lead to phase transformations. The

reversal of intensity of peaks (2 0 0) and (0 0 2) may be linked to the presence of ferroelasticity in tetragonal zirconia. Virkar and Matsumoto [49] demonstrated that the grinding process led to the formation of specific domains in the tetragonal grains—from which they inferred that domain switching under externally applied stress contributed to the toughening mechanism.

Therefore, the structural changes induced by sandblasting in this study could be explained either by local lattice distortions and/or by the emergence of a new phase (cubic 2) and/or by ferroelastic domain switching under stress. Other authors using Rietveld analysis on dental Y-TZP ceramics have also demonstrated the presence of the tetragonal phase and two cubic phases with slightly different lattice parameters [26,50,51]. The classic stress-induced phase transformation toughening of zirconia is the transformation of metastable tetragonal crystallites into their monoclinic isomers thus inducing compressive stresses at the crack tip. From our quantitative XRD performed at 30° (2θ) incidence angle, only minor amounts (< 5%) of monoclinic phase were detected after sand-blasting with 30 μm silica-coated alumina particles and could therefore not be properly fitted with the Rietveld procedure. Standard Bragg–Brentano XRD at 30° incidence angle with a depth penetration of 5 μm lacks reliability for monoclinic contents less than 5% [30]. Alternatively, grazing angles of 1–5° (2θ) would have demonstrated the stress-induced phase transformations more accurately. In effect thanks to a reduced penetration of the incident beam, these rays generate a higher surface-to-bulk signal ratio and indicate an increased quantity of *m* phase [30].

An additional explanation for the improvement of the fatigue limits after sandblasting is the “smoothing” of the surface’s machining grooves, which may act as stress concentrators. Although the measured roughness (Ra value) increased after CoJet sandblasting, the amount of abraded zirconia material of approximately 3 μm [52] provided a more homogeneous surface, rounding off machining grooves or eliminating these as illustrated in Fig. 14. Indeed, the SEM image of Fig. 14 shows the original ground surface (left) followed by the sandblasted section of a Zeno Y-TZP (Wieland) bend bar from our study. The deep grooves present on the original ground surface tend to disappear completely after sandblasting 20 s with the CoJet Sand. The image on the right represents a 3D reconstruction of a Zeno Y-TZP (Wieland) surface topography showing the vertical loss after sandblasting (blue surface) compared to the originally ground surface (red). [52]

Roughening a restoration’s intaglio surface by airborne particle abrasion (i.e. sandblasting) of zirconia restorations is mandatory for a durable resin bond [53]. The present study showed that sandblasting yttria stabilized zirconia with 30 μm silica-coated alumina particles at 2.5 bar also improved the fatigue limits and survival probabilities of the Y-TZP materials tested. It can therefore be recommended as a last clinical step before cementation.

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Fig. 1.

Technical drawing of the 3 point-bending assembly used for fatigue testing. The specimen in the center is supported by lateral axes (rollers) incorporated in anti-torsion devices allowing rotation, thus compensating for minor unevennesses in the surfaces.

Fig. 2. Microstructure of the four Y-TZP ceramics (20,000×). The average grain size according to the 'Linear interceptive count method' was as follows: Zeno (ZW) (383 ± 47 nm); Everest (KV) (383 ± 47 nm); Lava (LV) 537 nm (± 47); Lava colored (LVB) 643 nm (± 61).

Fig. 3.

(a and b) *S-N* fatigue data for Lava (LV) (3M Espe), Lava colored (LVB) (3M Espe), Everest ZS (KV) (KaVo), Zeno (ZW) (Wieland) “as received” and “after CoJet sandblasting”. Fatigue limits at 10^6 cycles at 10 Hz in water were determined from trend lines using a least square linear regression fit. The fatigue limits “as received” were: LV = 720 MPa, LVB = 600 MPa, KV = 560 MPa, ZW = 470 MPa. The fatigue limits of “CoJet sandblasted” were: LV = 840 MPa, LVB = 788 MPa, KV = 645 MPa, ZW = 540 MPa. The initial strength for the “as received” was: LV = 854 ± 94 MPa, LVB = 749 ± 209 MPa (one outlier with major grinding flaw was responsible for dropping the mean initial strength), KV = 776 ± 31 MPa, ZW = 742 ± 39 MPa. The initial strength for the “after CoJet

sandblasting” was: LVs = 1282 ± 119 MPa, LVBs = 1077 ± 102 MPa, KV_s = 836 ± 135 MPa, ZW_s = 787 ± 100 MPa.

Fig. 4.

Cumulative probability of survival at 1 million cycles for all four zirconia materials. Dashed lines denote the sandblasted groups. All four materials demonstrated a definite shift to the right as sandblasting improved their probability of survival. The median (50% at 10^6 cycles) survival values for the “as received” and “CoJet sandblasted” groups were as follows: LV = 743 MPa, LVs = 908 MPa, LVB = 635 MPa, LVBs = 809 MPa, KV = 593 MPa, KVs = 676 MPa, ZW = 549 MPa, ZWs = 587 MPa. The increase for Zeno (ZW) (green—far left) was not statistically significant ($p = 0.295$).

Fig. 5.

On the left, $S-N$ fatigue data (left) for Lava (LV) “as received” (black, bottom) and “CoJet sandblasted” (LVs) (red, top). Fatigue limits at 10^6 cycles at 10Hz in water were 720 MPa “as received” and 840 MPa for “CoJet sandblasted” which represented an increase of 17%. On the right, Kaplan–Meier cumulative probability of survival plot at 1 million cycles for Lava “as received”(left) and “CoJet sandblasted” (right). A clear shift to the right is seen when sandblasting, thus improving the survival probability.

Fig. 6. Stereomicroscope views (32× and 57× magnification) of two broken halves of a Lava (3M Espe) fatigue-failed zirconia specimen. The mirrors (smooth surfaces) and the radiating hackle lines surround the failure origin located on the tensile surface of this specimen.

Fig. 7.

Processing flaws for Zeno (ZW, Wieland) located at- or near the tensile surface, which acted as stress concentrators during fatigue testing. These flaws were incorporated into the ceramic during cold pressing of the blanks. The number of cycles and stress level for each image were as follows: (A) “Sandblasted”, 501,550 cycles at 581 MPa; (B) “as received”, 900 cycles at 635 MPa; (C) “as received”, 51 cycles at 658 MPa; (D) “as received”, 1577 cycles at 548 MPa.

Fig. 8.

For the Lava and Everest ceramics, very few inherent processing flaws located at- or near the tensile surface acted as stress concentrators at the failure origin. (A) Subsurface pore (30 μm × 10 μm) on a Lava sandblasted specimen, which failed after 12,290 cycles at 840 MPa; (B) surface pore (10 μm × 10 μm) on a Lava colored sandblasted specimen which failed after 960 cycles at 611 MPa. (C) Small (10 μm × 15 μm) subsurface sintering dry spray agglomerate flaw on a Lava sandblasted specimen which survived 10^6 cycles at 951 MPa, then was monotonically fractured at 1489 MPa; (D) Everest sandblasted specimen demonstrating failure from a large grain due to critical grain growth acting as failure origin with a size of 20 μm × 60 μm. This specimen failed shortly after passing the minimum amount of fatigue cycles (50) to be validated as it failed after 53 cycles for a stress level of 747 MPa.

Fig. 9. Grinding and blank pressing failure flaws on the fatigue curve for ZW “as received” specimens. For specimens failing below 2000 cycles, the pressing flaws outnumbered the grinding flaws (4 vs. 10). After 2000 cycles, the grinding flaws prevailed (8 vs. 4).

Fig. 10.

Examples of critical grinding flaws located at the tensile surface and acting as stress concentrators during fatigue testing. (A) Zeno ZW (Wieland), failure after 2473 cycles at 532 MPa; (B) Zeno ZW (Wieland), failure after 89 cycles at 524 MPa; (C) Everest sandblasted KV (KaVo), failure after 233 cycles at 723 MPa; (D) Zeno ZW (Wieland), failure after 591 cycles at 519 MPa; (E) Lava colored LVB (3M Espe), failure after 21,458 cycles at 602 MPa; (F) Lava colored sandblasted LVB (3M Espe), failure after 2576 cycles at 850 MPa.

Fig. 11. Surface characteristics of the machined specimens for all four materials. (A) Zeno (Wieland) showing a combination of longitudinal, transverse and oblique grinding marks. (B) Everest (KaVo) reshaped from the sides leaving a central portion with transverse machining and a smoother peripheral surface made of oblique and longitudinal grinding marks. Lava (C) and Lava colored (D) (3M Espe) showing predominantly longitudinal machining, with some deeper grooves in the surface.

Fig. 12.

XRD for Everest (KV) “as received” (bottom) and after “CoJet sandblasting” (top). The sandblasted profile (top) displayed an asymmetrical broadening at the (1 0 1), (2 0 2) and (1 1 3) peaks and the reversal of intensities of peaks (0 0 2) and (2 0 0). A slight (below 5%) $t \rightarrow m$ transformation was also noted at $\sim 28^\circ 2\theta$.

Fig. 13. Clinical 3Y-TZP Lava bridge framework viewed under stereomicroscope as well as under SEM. The multidirectional grinding marks are typical for CAD–CAM machining.

Fig. 14.

SEM of original ground surface (left) followed by the sandblasted section of a Zeno Y-TZP (Wieland) bend bar from this study. The deep grooves present on the original ground surface tend to disappear completely after sandblasting 20 s with the CoJet Sand. The image on the right represents a 3D reconstruction of a Zeno Y-TZP (Wieland) surface topography showing the vertical loss after CoJet sandblasting (blue surface) compared to the originally ground surface (red).

Table 1

Y-TZP materials selected for fatigue testing.

Brand	Materials	Manufacturer
LAVA (LV)	3Y-TZP	3M Espe, Seefeld, D
LAVA colored (LVB)	3Y-TZP	3M Espe, Seefeld, D
EVEREST ZS (KV)	3Y-TZP	KaVo Dental GmbH, Biberach, D
ZENO Zr (ZW)	3Y-TZP	Wieland, Dental, Pforzheim, D

Table 2

Density and roughness (Ra) in μm and standard deviation (SD) of the four zirconia materials.

Zirconia groups	Density (g/cm³) (SD)	Roughness (μm) (Ra) (SD)
Lava (LV)	6.09 (0.02)	0.23 (0.01)
Lava sandblasted (LVs)		0.26 (0.01)
Lava colored (LVB)	6.12 (0.003)	0.18 (0.01)
Lava colored sandblasted (LVBs)		0.27 (0.02)
Everest (KV)	6.08 (0.01)	0.75 (0.58)
Everest sandblasted (KVs)		0.71 (0.55)
Zeno (ZW)	6.09 (0.004)	0.30 (0.06)
Zeno sandblasted (ZWs)		0.35 (0.04)

Table 3

Static 3-point bending of mirror polished bars immersed in water. Weibull's characteristic strength (σ_0), Weibull's modulus (m), and corresponding confidence intervals (CI), at 95% confidence levels.

Material	N	Weibull σ_0 (MPa)	CI σ_0 (95%)	Weibull m	CI m (95%)
Lava (LV)	10	1176	1135–1218	18.5	11.2–30.6
Lava colored (LVB)	9	1072	999–1150	9.8	5.9–16.4
Everest ZS (KV)	10	1115	1049–1186	10.7	6.5–17.5
Zeno Zr (ZW)	10	728*	696–761	14.7	9.1–23.9

The Zeno ceramic was significantly inferior (*) in σ_0 but no differences were evidenced in the Weibull m parameters.

Kaplan–Meier median percentiles survival stress (in MPa) with 95% confidence intervals for 1 million fatigue cycles and two surfaces: “as received” (control) vs. CoJet sandblasting. Superscripts refer to the Holm–Sidak statistical analysis.

Table 4

N cycles, 10 Hz, in H ₂ O	Y-TZP surface	ZW (Zeno Wieland)	KV (Everest (KaVo))	LVB (Lava colored (3M Espe))	LV (Lava white (3M Espe))
10 ⁶	ctr	549 (543–555) ^a	593 (579–607) ^a	635 (578–692) ^b	743 (729–757) ^b
	CoJet	587 (545–629) ^a	676 (655–697) ^b	809 (787–831) ^c	908 (840–976) ^c

Table 5

Distribution of flaws in the 'as received' specimens as observed by SEM.

Material	Samples (N) observed (SEM)	Blank press flaw	Grinding flaw	Glass inclusion flaw
ZW as received	26/30	15	11	0
KV as received	23/30	0	22	1
LV as received	20/30	0	19	1
LVB as received	24/29	1	21	2

Table 6

Rietveld analysis for Everest, Lava, Lava colored and Zeno.

Sample	χ^2	Tetragonal phase		Cubic 1 phase		Cubic 2 phase	
		wt. %	<i>a</i> and <i>c</i> (Å)	wt. %	<i>a</i> (Å)	wt. %	<i>a</i> (Å)
Everest (KV)	2.56	72 (2)	3.606 (2), 5.180 (2)	28 (2)	5.129 (2)	–	–
Everest (KV) + CoJet	1.90	53 (3)	3.605 (5), 5.178 (7)	–	–	47 (3)	5.156 (8)
Lava (LV)	2.32	87 (2)	3.606 (1), 5.180 (1)	13 (2)	5.132 (1)	–	–
Lava (LV) + CoJet	2.02	62 (2)	3.605 (4), 5.176 (6)	–	–	38 (2)	5.162 (6)
Lava colored (LVB)	1.61	76 (1)	3.605 (1), 5.180 (1)	24 (1)	5.131 (2)	–	–
Lava colored (LVB) + CoJet	2.13	64 (3)	3.606 (5), 5.173 (7)	–	–	36 (3)	5.159 (8)
Zeno (ZW)	1.46	92 (1)	3.606 (1), 5.178 (2)	8 (1)	5.131 (1)	–	–
Zeno (ZW) + CoJet	1.88	82 (2)	3.605 (5), 5.175 (7)	–	–	18 (2)	5.167 (8)