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THE FRACTURE AND FATIGUE OF SURFACE-TREATED TETRAGONAL ZIRCONIA (Y-TZP) DENTAL CERAMICS

PRELOM IN UTRUJENOST POVRŠINSKO OBDELANE TETRAGONALNE (Y-TZP) DENTALNE KERAMIKE

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The effects of dental grinding and sandblasting on the biaxial flexural strength of Y-TZP ceramics containing the mass fraction of 3 % yttria were evaluated. Dental grinding at high rotation speed lowers the mean strength under static loading and the survival rate under cyclic loading. Sandblasting, in contrast, may provide a powerful tool for surface strengthening also resulting in a substantially higher survival rate under cyclic loading. Fractographic examination of ground specimens revealed that failure originated from radial cracks extending up to 50 μm from the grinding groves into the bulk of the material. However, no evidence of grinding-induced surface cracks could be obtained by SEM analysis of the ground samples, prepared by a standard bonded-interface technique. Sandblasting, in contrast, introduces lateral cracks, which are not detrimental to the strength of Y-TZP ceramics. The "medical-grade" Y-TZP ceramics also containing 0.25 % of dispersed alumina used in this work exhibited full stability under hydrothermal conditions.

Key words: tetragonal zirconia; dental grinding; sandblasting; fracture origin; fatigue

Ocenjen je bil vpliv zobnih brušenj in peskanja na dvoosno upogibno trdnost keramike Y-TZP z molskim deležem itrijevega oksida 3 %. Zobno brušenje pri veliki hitrosti vrtenja zmanjša trdnost pri statični obremenitvi in trajnostno dobo pri ciklični obremenitvi. Nasprotno pa je peskanje lahko učinkovit način za utrditev površine, ki pomembno poveča tudi trajnostno dobo pri ciklični obremenitvi. Fraktografsko opazovanje brušenih preizkušancev je pokazalo, da se je prelom začel iz radialnih razpok v globino do 50 μm iz dna brusilnih žlebov v notranjost preizkušanca, čeprav pri SEM pregledu ni bila odkrita nobena površinska brusilna razpoka na brušenih preizkušancih, pripravljenih po standardni površinsko vezani tehniki. Peskanje nasprotno od brušenja ustvari lateralne razpoke, ki ne vplivajo na trdnost keramike Y-TZP. Tetragonalna (Y-TZP) keramika za medicinske namene, ki vsebuje tudi 0,25 % dispergiranega aluminijevega oksida in je bila uporabljena v tem delu, je bila popolnoma stabilna v hidrotermalnih razmerah.

Ključne besede: tetragonalni cirkonijev oksid, zobno brušenje, peskanje, začetek razpoke, utrujenost

1 INTRODUCTION

Yttria partially stabilized tetragonal zirconia (Y-TZP) has become increasingly popular as an alternative high-toughness core material in dental restorations because of its biocompatibility, acceptable aesthetics and attractive mechanical properties. Compared to other dental ceramics, the superior strength, fracture toughness and damage tolerance of Y-TZP are due to a stress-induced transformation toughening mechanism operating in this particular class of ceramics [1]. Y-TZP is currently used as a core material in full-ceramic crowns and bridges, implant superstructures, orthodontic brackets and root dental posts [2–5]. Like most technical ceramics, zirconia dental restorations are produced by dry- or wet-shaping of ceramic green bodies which are than sintered to high density. For the material's selection and microstructural design the following two criteria should be taken into consideration: the damage tolerance upon mechanical surface treatment and the aging behavior in an aqueous environment. Dental grinding is involved in reshaping and the final adjustment of the prosthetic work, whereas sandblasting is commonly used to improve the bond between the luting agent and the prosthetic work. Because Y-TZP ceramics exhibit a stress-induced transformation, the surface of the mechanically treated prosthetic work is expected to be transformed into the monoclinic form, i.e. constrained, and also damaged. Under clinical conditions, where dental restorations are exposed to thermal and mechanical cycling in a chemically active aqueous environment over long periods, these grinding- and impact-induced surface flaws may grow to become stress intensifiers, facilitating fracture at lower levels of applied stress. Furthermore, with prolonged time under clinical conditions the metastable tetragonal zirconia may start transforming spontaneously into the monoclinic structure [6]. This transformation is diffusion-controlled and is accompanied by extensive microcracking, which ultimately leads to strength degradation [7]. Therefore, extensive research work was undertaken to evaluate the effects of mechanical surface treatment and aging on the strength and reliability of various Y-TZP ceramics.

In our previous studies [8,9] we have shown that dental grinding using a coarse-grit diamond burr at a high rotation speed lowers the mean strength and reliability, whereas sandblasting improves the mean strength, at the expense of somewhat lower reliability. The fine-grained materials exhibited higher strength after sintering, but they were less damage tolerant upon grinding than tougher, coarse-grained materials. Standard grade 3Y-TZP ceramics were more susceptible to low-temperature degradation than a special, corrosion resistant 3Y-TZP grade also containing a small amount of dispersed alumina. Besides, no grain-size dependence of the diffusion-controlled transformation was observed with this material. Based on these results, coarse-grained zirconia containing a small amount of alumina was suggested for dental applications.

Here we report on the fracture and fatigue of surface-treated tetragonal zirconia (Y-TZP) dental ceramics. Fracture mechanics was used to calculate the effective length of mechanically induced surface flaws acting as the stress concentrators, relative to the depth of the stress-induced surface compressive layer which contributes to strengthening. The results were verified by a conventional fractographic examination as well as by SEM analysis of surface-treated samples, which were prepared by a standard bonded-interface technique.

2 EXPERIMENTAL WORK

Disc-shaped specimens ((15.5 ± 0.03) mm in diameter ad (1.5 ± 0.03) mm thick) were fabricated from a commercially available ready-to-press Y-TZP powder (TZ-3YSB-E, Tosoh, Japan) containing the mass fraction of yttria 3 % in the solid solution and a 0.25 % alumina addition to suppress the t-m transformation during aging, by uniaxial dry pressing and pressureless sintering in air for 4 h at 1450 °C and 1550 °C, respectively.

After firing, the top surface of the specimens was submitted to a different surface treatment. A coarse grit (150 μm) and a fine grit (50 μm) diamond burr were chosen for dry and wet surface grinding, in order to simulate clinical conditions. The grinding load of about 100 g was exerted by finger pressure, the grinding speed was 150,000 r/min. For sandblasting, discs were mounted in a sample holder at a distance of 30 mm from the tip of the sandblaster unit, equipped with a nozzle of 5 mm in diameter. Samples were sandblasted for 15 s with 110 μm fused alumina particles at 4 bar. Before and after each surface treatment the samples were analyzed by XRD, using CuKα radiation. The relative amount of transformed monoclinic zirconia on the specimens' surfaces was determined according to the method of Garvie and Nicholson [10]. The thickness of the transformed surface layer of surface-treated samples was calculated using the x-ray determination method [11]. Although this method yields conservative values, it can be used to compare the influence of various surface treatments on the thickness of the surface compressive layer.

Aging of pristine and mechanically treated materials in an aqueous environment was performed under isothermal conditions at 140 °C for 24 h. After aging, specimens were analysed by XRD for phase composition.

Biaxial flexural strength measurements were performed according to ISO 6872 at a loading rate of 1 mm/min. Surface-treated specimens were fractured with the surface treated side under tension. The load to failure was recorded for each disc and the flexural strength was calculated using the equations of Wachtman et al.[12]. The variability of the flexural strength values was analyzed using the two-parameter Weibull distribution function.

Cyclic loading experiments were performed using an Instron Ltd, Model 8871 machine. The load varied from 50 N to 850 N at a freqency of 15 Hz. After 10^6 cycles the specimens were "statically" loaded to fracture. For specimens which failed before one milion cycles The number of cycles to failure was registered.

After biaxial flexural strength measurements the fracture surfaces were examined by SEM. The existence of grinding- and sandblasting-induced sub-surface flows was evidenced by SEM analysis of polished interfaces perpendicular to the ground and sandblasted surface, respectively. For this examination, specimens were prepared using a standard bonded-interface technique, as described elsewhere [13].

3 RESULTS AND DISCUSSION

The main characteristics of the sintered materials are listed in **Table 1**. The relative density of sintered specimens exceeded 99 % of the theoretical value and they were 100 % tetragonal. An SEM micrograph of the sintered material, showing equiaxed grains with the mean size of 0.57 μm, is represented in **Figure 1**.

Table 1: Sintering conditions and main characteristics of sintered ceramics
Tabela 1: Pogoji sintranja in osnovne značilnosti sintrane keramike

Sintering conditions	Mean grain size d/μm	Flexural strength MPa (SD)	K_{Ic} MPa m$^{1/2}$ (SD)
1450 °C/4 h	0.51	1080 (75)	5.08 (0.10)
1550 °C/4 h	0.59	990 (111)	5.18 (0.12)

Tosoh, Tokyo, Japan

During dental grinding, tens of μm of material were removed by a single pass as the burr was moved back and forth across the surface and the process was always accompanied by extensive sparking. During sandblasting about 60 μm of material was uniformly removed but sparks were not observed during this operation. Microscopic examination of the ground and sandblasted samples revealed that in both cases the materials surface

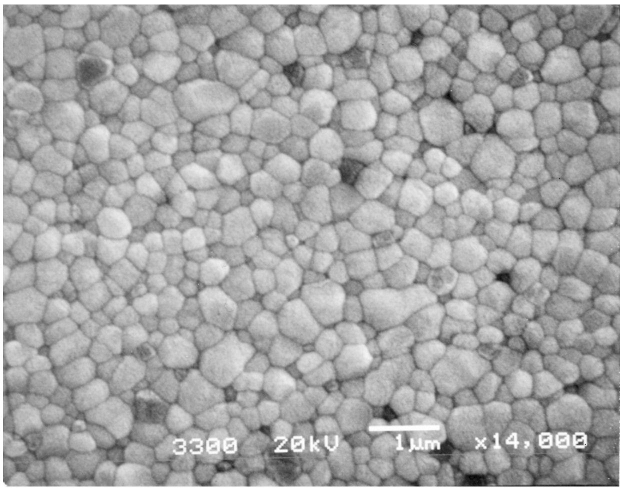

Figure 1: SEM micrograph showing the microstructure of sintered tetragonal zirconia
Slika 1: SEM-posnetek, ki prikazuje mikrostrukturo sintranega cirkonijevega oksida

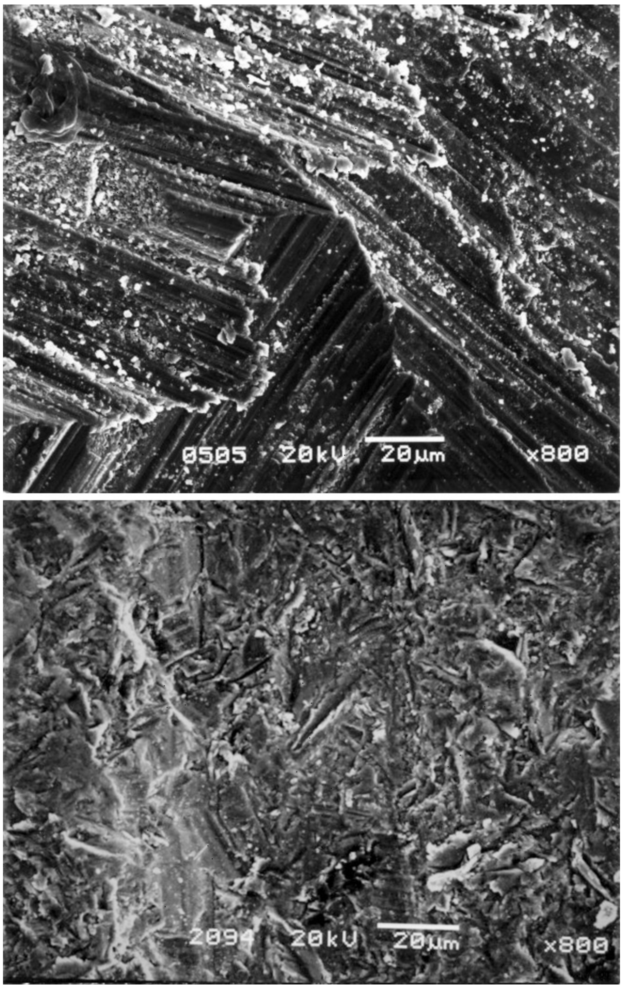

Figure 2: SEM micrographs showing Y-TZP surface morphology after: A) dry grinding using 150 µm diamond burr and B) sandblasting
Slika 2: SEM-posnetka, ki prikazujeta morfologijo površine keramike Y-TZP po: A) suhem brušenju z diamantnim brusom 150 µm in B) po peskanju

was in part plastically deformed (**Figure 2**). The depth of the intersecting grinding grooves and parallel grit scratches, representing the most characteristic feature of the ground surface morphology, varied with the diamond grit size. The eroded surface was wrinkled with sharp, randomly oriented scores, and surface pits were readily observed on an otherwise plain sandblasted surface. In spite of high stresses during grinding the amount of transformed zirconia on the ground surfaces was almost negligible, and so was the transformed zone depth, as calculated from the relative amounts of the monoclinic phase. It is assumed that during grinding the locally developed temperatures exceeded the m->t transformation temperature and the reverse transformation occurred. Higher amounts of the monoclinic zirconia, about 15–17%, were detected on sandblasted samples, which yielded the transformed-zone depth values ranging from 0,3 µm to 0,5 µm. It is interesting to note that these values roughly correspond to the mean grain size of the sintered ceramics.

The mean values of biaxial flexural strength and the respective standard deviations are graphically represented in **Figure 3**. Dental grinding evidently lowered the mean strength, whereas sandblasting provided a powerful tool for strengthening. The counteracting effect of dental grinding and sandblasting on flexural strength can be explained by considering two competing factors influencing the strength of surface treated Y-TZP ceramics: residual surface compressive stresses, which contribute to strengthening, and mechanically induced surface flaws, which cause strength degradation [8].

Since almost no monoclinic zirconia was detected on the ground specimens, the contribution of the grinding-induced strengthening must have been negligible, regardless of grinding conditions used and the material tested. The strength of ground materials is thus mainly

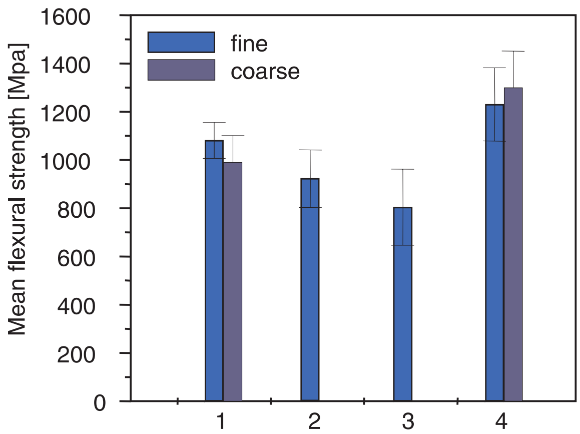

Figure 3: Mean biaxial flexural strength values for as-sintered and surface treated Y-TZP ceramics. 1 – As sintered, 2 – Dry ground (50 µm diamond burr), 3 – Dry ground (150 µm diamond burr), 4 – Sandblasted. Error bars represent one SD from the mean.
Slika 3: Povprečna dvoosna upogibna trdnost sintrane in površinsko obdelave keramike Y-TZP. 1 – sintrano, 2 – suho brušeno (diamantni brus 50 µm), 3 – suho brušeno (diamantni brus 150 µm), 4 – peskano. Območje raztrosa prikazuje eno SD od povprečja.

determined by the critical defect size to initiate failure, which can be estimated using the Griffith strength relation [14]

$$\delta_f = \frac{1}{\varphi} \cdot \frac{K_{IC}}{\sqrt{c_{cr}}} \quad (1)$$

where δ_f is the fracture stress, φ is a geometric constant (= $2/\pi^{1/2}$ for surface line cracks), K_{IC} is the fracture toughness and c_{cr} is the critical defect size to initiate failure. Calculated c_{cr} values for sintered coarse-grained specimens before and after dry grinding using 150 µm and 50 µm grit burr were 17.3, 31.0 µm and 24.1 µm, respectively. Since Eq. (1) does not take into account any of the residual surface stresses that may exist in the material, the calculated c_{cr} values should be regarded as the effective length of strength-controlling defects, which would result in an equivalent strength of the material without any residual surface stresses. Surface grinding increases the effective critical defect size, presumably by generating radial surface cracks. A fractographic examination of the ground specimens indeed revealed that failure originated from radial cracks extending several tens of µm (up to 50 µm) from the grinding grooves into the bulk of the material (**Figure 4**). However, no evidence of grinding-induced surface cracking could be obtained by SEM examination of a polished interface perpendicular to the ground surface (**Figure 5**). This observation is in agreement with recently published results by Xu et al [15], who reported on a noticeable grit size dependence of strength degradation upon machining of Y-TZP, but they could not find any evidence of grinding-induced surface cracking. Since radial cracks, which are readily seen in fracture surfaces, were not formed during grinding, they must have been initiated and extended from a grinding groove during loading until they reached the critical length for failure initiation.

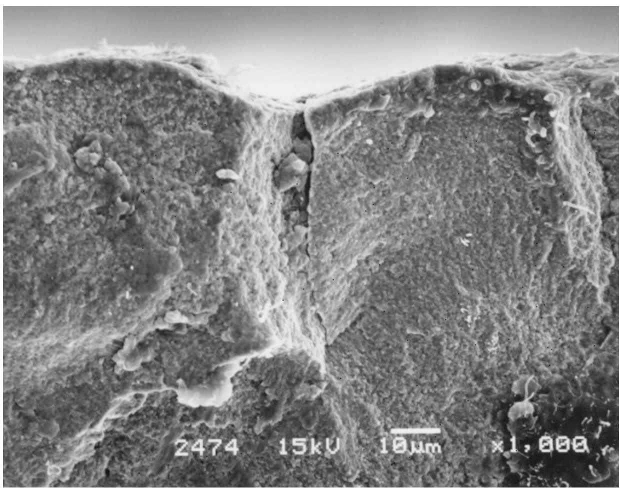

Figure 4: SEM micrograph of the fracture surface of fine grained dry ground Y-TZP

Slika 4: SEM-posnetek prelomne površine fino suho brušene Y-TZP

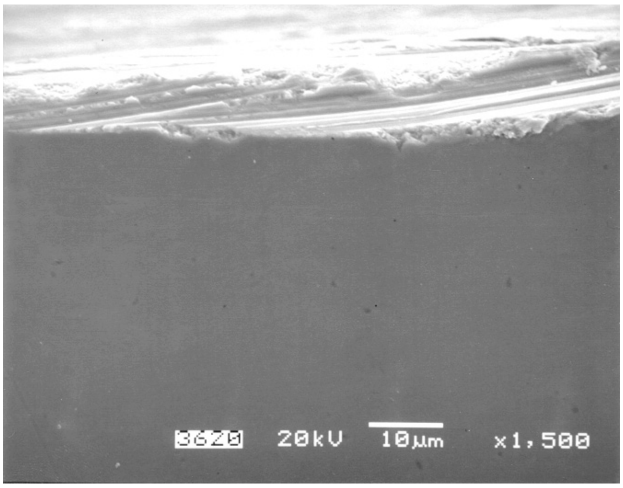

Figure 5: SEM micrograph of a polished interphase perpendicular to the ground (150 µm grit) of a fine grained Y-TZP

Slika 5: SEM-posnetek polirane površine, pravokotne na brušeno (zrno 150 µm) fino zrnato Y-TZP

Subcritical crack growth from a grinding groove during cyclic loading resulted in the lowest survival rate during fatigue experiments, whereas the strength of "survived" specimens was nearly the same as that of the material which was not subjected to cyclic loading.

In contrast to grinding, sandblasting is capable of transforming a larger amount of zirconia in the surface of Y-TZP ceramics indicating lower temperatures during this operation. Surface flaws, which are introduced by sandblasting, do not seem to be strength determining, otherwise the strength of the material would have been reduced instead of being increased. Since lateral crack chipping is the most prevalent mechanism involved in the erosive wear of ceramics, lateral cracks could be expected in these samples, which was later confirmed by

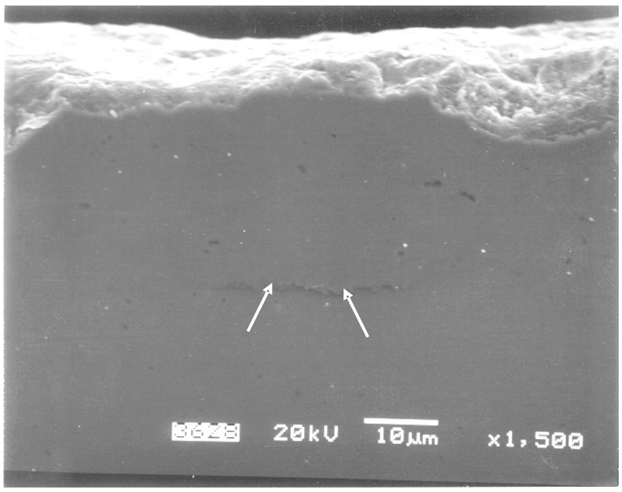

Figure 6: SEM micrograph of a polished interphase perpendicular to the sandblasted surface of a fine-grained Y-TZP, showing lateral crack chipping

Slika 6: SEM-posnetek polirane površine, pravokotne na peskano ploskev fino zrnate Y-TZP, ki prikazuje lateralno luščilno razpoko

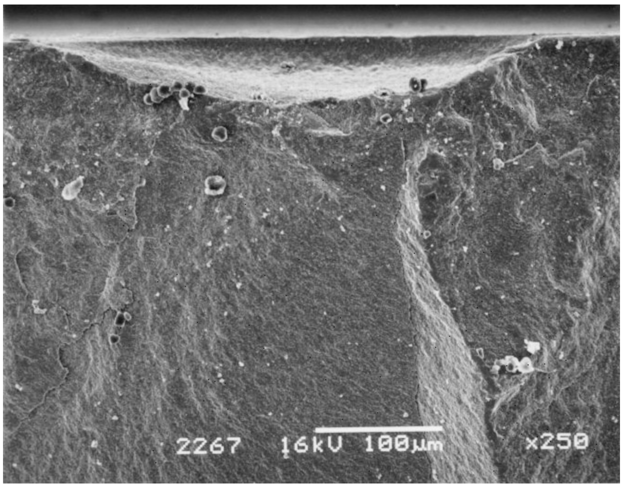

Figure 7: SEM micrograph of a fracture surface of fine-grained dry ground and sandblasted Y-TZP. Failure originated from a 50 µm deep surface pit

Slika 7: SEM-posnetek prelomne površine finozrnate suho brušene in peskane Y-TZP. Začetek preloma je v 50 µm globoki površinski zajedi.

microscopic examination using a bonded-interface technique (**Figure 6**). Fractographic examination of sandblasted samples confirmed that the failure of these samples was initiated from a lateral crack linked to subsurface cracks (**Figure 7**). It seems that sandblasting introduces surface flaws, which are not detrimental to the strength of Y-TZP ceramics statically loaded to fracture. However, under clinical conditions these impact flaws may grow to become stress intensifiers, causing accidental failure at lower levels of applied stress.

After autoclaving at 140 °C for 24 h, traces of the monoclinic zirconia were identified on the surface of sintered specimens, but the strength degradation has not yet occurred. The same observation was made with the ground and sandblasted specimens.

4 CONCLUSIONS

The surface grinding using a coarse-grit diamond burr at a high rotation speed lowers the mean strength of tetragonal zirconia Y-TZP ceramics, whereas sandblasting provides a powerful method for surface strengthening. The counteracting effect of dental grinding and sandblasting was explained in terms of two competing factors influencing the strength of surface treated 3Y-TZP ceramics: residual surface compressive stresses, which contribute to strengthening, and mechanically induced surface flaws, which cause strength degradation.

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