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The effect of surface treatments on dental zirconia: An analysis of biaxial flexural strength, surface roughness and phase transformation

Michael G. Botelho[a,*], Sachin Dangay[b], Kaimin Shih[c], Walter Y.H. Lam[a]

[a] Prosthodontics, Faculty of Dentistry, The University of Hong Kong, Hong Kong, China
[b] Private Practice, New Delhi, India
[c] Department of Civil Engineering, Faculty of Engineering, The University of Hong Kong, Hong Kong, China

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ABSTRACT

Objectives: This study investigates the effect of selected surface finishing techniques on the biaxial flexural strength, surface roughness and phase transformation of a zirconia dental restorative material.
Materials and methods: Fully-sintered zirconia discs (ø19.5 mm × 0.85 mm) were treated on one side with a single or a combination of the following treatments: diamond and/or tungsten-carbide burs without water coolant in an air-turbine handpiece, air-particle abrasion, rubber-point polishing in a contra-angle handpiece, or no treatment (control). Biaxial flexural strength (BFS) (eleven groups, n = 10) was determined using a universal testing machine and surface roughness (thirteen groups, n = 6) was assessed using a profilometer. Results were analysed using one-way ANOVA and Student-Newman-Keuls Post-hoc test ($\alpha = 0.05$) with Bonferroni correction. Specimens were observed under scanning electron microscopy (SEM) and x-ray diffraction (XRD) for their microstructure and crystalline phases respectively.
Results: Grinding with diamond burs did not weaken zirconia ($p > 0.0045$) but produced rougher surfaces than the control group ($p < 0.0038$). Tungsten-carbide burs smoothened diamond ground specimens ($p < 0.0038$) for both grits of diamond. Specimens ground by tungsten-carbide burs have significantly reduced mean BFS ($p < 0.0045$) by up to two-thirds and SEM revealed fine surface cracks. Air-particle abrasion restored the mean BFS of tungsten-carbide ground specimens to control levels ($p > 0.0045$) and surface cracks were not observed. Phase transformation was not detected by XRD.
Conclusions: Dental zirconia ground dry with tungsten-carbide burs has a significantly reduced BFS and a smooth but defective surface. These defects may be removed and BFS restored by air-particle abrasion.
Clinical significance: The use of tungsten-carbide burs for grinding dental zirconia should be cautioned. Diamond grinding does not weaken zirconia but requires further polishing.

1. Introduction

The use of zirconia-based ceramics in restorative and implant dentistry has grown significantly in recent years due to their desirable aesthetics, high biocompatibility and superior mechanical properties [1–4]. Dental zirconia is usually produced by stabilizing zirconia with alloying metal oxides such as yttrium oxide (Y_2O_3, 3 mol%) to produce yttria-tetragonal zirconia polycrystal (3Y-TZP). At room temperature, this stabilization maintains the tetragonal phase of 3Y-TZP by controlling the tetragonal (space group D_{4h}^{15} or $P4_2/nmc$) to monoclinic (space group C_{2h}^{5} or $P2_1/c$) ($t \rightarrow m$) transformation [5]. When 3Y-TZP is under stress, $t \rightarrow m$ transformation occurs which is associated with a local volumetric increase (~4.5%) which in turn has the effect of compressing the crack defects thereby preventing further propagation and so increases the flexural strength of the zirconia [6–8]. However, this process may destroy the phase integrity of zirconia and may contribute to early ageing or low temperature degradation in the presence of water, leading to surface degradation and a reduction in strength [4,9].

Dental zirconia restorations have been traditionally veneered with low-fusing feldspathic porcelain. However, a relatively high incidence of chipping and fracture of porcelain layer has been reported among these restorations [10,11]. With the development of zirconia, monolithic zirconia restorations have been suggested to address this complication and thus have grown in popularity [12]. Insertion of dental restorations in patients' mouths may often require grinding which leaves a rough and defective surface [13]. This may have a negative impact on strength and wear characteristics [14,15]. However, there is

* Corresponding author at: Prosthodontics, Faculty of Dentistry, The University of Hong Kong, 34 Hospital Road, Prince Philip Dental Hospital, Sai Ying Pun, Hong Kong, China.
E-mail address: botelho@hku.hk (M.G. Botelho).

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conflicting information on the effects that surface treatments have on the strength of zirconia materials with some reports showing a decrease in strength [16–22] and others an increase [20–22]. While grinding may create defects and weaken the zirconia [23], air particle alumina abrasion [22] and sometimes grinding [20,22] may generate stresses which can trigger a $t \rightarrow m$ transformation and therefore increase local strength by preventing potential growth of crack [20–22,24]. On the other hand, early aging or excessive temperature increase during grinding may cause reversal of transformation $m \rightarrow t$. The cracks may then propagate easily and reduced the strength of zirconia [25–27]. The final strength of zirconia is hence determined by the volume percentage of transformed zirconia (m), the grinding severity, and the locally developed temperatures [22].

Moreover, the surface roughness of zirconia may affect the surface stress state and indirectly affect ageing which starts at the surface [28]. Dental zirconia should also be well polished to reduce antagonist wear [29]. Various polishing protocols have been suggested to finish dental ceramic materials and these often involve the use of diamond burs of different grit sizes and rubber points or finishing discs. The effects of dental tungsten carbide burs for surface smoothing zirconia does not appear to have been investigated fully. The aim of this study was to investigate the effect of selected surface finishing techniques on the biaxial flexural strength and surface roughness of a zirconia dental restorative material. The null hypotheses were that there was no difference in (a) mean biaxial flexural strength and (b) mean surface roughness between the experimental groups and the controls.

2. Materials and methods

2.1. Preparation of specimens

Cylindrical blanks of partially-sintered yttria-stabilized dental zirconia (Cercon Base, 25 mm diameter; Degudent, Dentsply, Germany) were found to have around 22% sintering shrinkage in diameter. They were cut wet with an annular saw (Microslice 2; Ultratech, USA) to produce discs of 1.1 mm thick and defective discs were discarded. Discs were sintered (Cercon heat furnace; Degudent, Dentsply, Germany) at 1350 °C for 6 h according to the manufacturer's recommendations and were allowed to cool to room temperature. No glazing or staining was performed. After sintering, the diameter of zirconia discs was reduced to 19.5 mm. Discs of 0.85 mm (± 0.05 mm) thick were selected by a Vernier calliper (530-312; Mitutoyo, Japan). From 220 discs produced for trial and for experimental use, 110 discs were selected (11 groups × 10 per group) for testing the biaxial flexural strength (BFS) and 78 discs were selected (13 groups × 6 per group) for testing the surface roughness. An extra set of specimens (11) was prepared for X-ray diffractometer (XRD) and scanning electron microscopy (SEM) analysis in the BFS test. Discs were randomly divided among groups. All specimens were cleaned in an ultrasonic water bath for 30 s before any surface treatments.

2.2. Grinding

An air-turbine handpiece (TA-98L, Synea HS, W&H, Austria) and a contra-angle handpiece (WA-56LT, Synea LS, W&H, Austria) were operated at full speed (360,000 and 40,000 rpm respectively). Each disc was ground uniformly on one side by a single operator with the cutting side of the bur (Table 1) oriented parallel to the disc surface and the bur moved in single direction across the surface for 10 s using manual clinical force. The force involved has been estimated to be around 100 g [30]. Handpieces were run for 1 min before the treatment to prevent oil contamination [17]. All specimens were ground dry to observe the greatest potential adverse effects associated with grinding and the bur was discarded after every four specimens to maintain similar cutting efficiency [20,23].

Table 1
Burs and rubber points used for the surface grinding and polishing.

Air-turbine burs		Model	Manufacturer
Diamond	Coarse 120 µm grit	6847 KR.314.018 (ISO # 806 314 546534 018)	Komet, Germany
	Fine 25 µm grit	6847 KR.314.016 (ISO # 806 314 545504 016)	Komet, Germany
Tungsten carbide	30 blades	H246UF.314.009 (ISO # 500 314 496031 009)	Komet, Germany
	8 blades	H283.314.010 (ISO # 500 314 289072 010)	Komet, Germany
Contra-angle rubber points			
Brownie and Greenie		403 and 404, Brownie/ Greenie/Supergreenie (ISO # 030)	Shofu, Japan
Porcelain polishing rubber points		Ultra 256A and Ultra II 259A, Ceramisté (ISO # 060)	Shofu, Japan

Table 2
Surface treatment procedures performed on fully sintered zirconia discs including specimen's code descriptors for biaxial flexural strength testing.

Group	Surface grinding		Air particle abrasion	Specimen code
	Diamond	Tungsten carbide		
A	D			D
B	D		A	DA
C	D	Tf		DTf
D	D	T		DT
E	D	T	A	DTA
F	Df			Df
G		T		T
H		T	A	TA
I		Tf		Tf
J		Tf	A	TfA
K				C (Control)

T = 8 blades tungsten carbide, Tf = 30 blades tungsten carbide, D = coarse diamond, Df = fine diamond, A = air particle abrasion (50 µm diameter).

2.3. Biaxial flexural strength (BFS)

For the biaxial flexural strength testing, eleven groups (n = 10) were treated with grinding, air particle abrasion and including a *control* group (no surface treatment) (Table 2). Groups *A* to *E* were ground with a coarse diamond bur and groups *G* to *J* with 8 or 30 blades finishing tungsten carbide burs. Group *F* was ground with a fine diamond and groups *C*, *D* and *E* were ground with both diamond and tungsten carbide burs. Groups *B, E, H* and *J* were further abraded by air particles of 50 µm diameter (Vacumat 300; Vita Zahnfabrik, Germany) perpendicularly from a distance of 20 mm at a pressure of 50 Psi for 10 s (Shofu Pen-Blaster; Shofu, Japan). Group *K* was the *control*.

After 24 h dry storage, specimens were tested for BFS using a piston of 1.4 mm diameter on a three-ball testing design in a universal testing machine (ElectroPuls E3000; Instron, USA). Balls were of 2 mm diameter and 6 mm from the centre of the platform. Specimens were placed with treatment side down on the assembly and loaded to failure with 1-kN load cell at a crosshead speed of 1 mm/min. The testing was performed following ISO 6872:2008 [31]. The yielding loads at fracture P (Newton) were recorded and the corresponding BFS σ (MPa) were then calculated by the following equation:

$$\sigma = \frac{-0.2387P(X - Y)}{b^2}$$

where $X = (1 + \nu)\ln(\frac{r_2}{r_3})^2 + \left(\frac{1-\nu}{2}\right)\left(\frac{r_2}{r_3}\right)^2$; $Y = (1 + \nu)(1 + \ln\left(\frac{r_1}{r_3}\right)^2) + (1 - \nu)(\frac{r_1}{r_3})^2$; b is the specimen thickness at fracture origin (mm); ν is Poisson's ratio (0.25 for dental ceramics); r_1 is the radius of support circle (mm); r_2 is the radius of loaded area (mm); r_3 is the