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Published online: 22 Jun 2017.

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Fatigue behaviours of the zirconia dental restorations prepared by two manufacturing methods

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ABSTRACT

The fatigue behaviours were evaluated on a novel zirconia dental restoration known as self-glazed zirconia (SG), prepared by a precision additive 3D gel deposition approach, compared with a conventional zirconia (CZ) restoration, shaped by CNC milling of zirconia blanks made by cold isostatic pressing. Eight fixed partial dentures made by each method were subjected to fracture test, without or with the application of 5-million fatigue cycles, respectively. The processing defects, grain size, and t–m phase transformation were examined by SEM and XRD. The results revealed that the fracture force of the SG restorations was higher than that of the CZ restorations in both cases, which ascribed to the fact that more voids and larger grains in the conventional versus in the SG restorations. The t–m phase transformation was observed only on the fracture surfaces of both materials subjected to fatigue test. Both zirconia restorations meet the clinical requirement.

Introduction

Missing or defective teeth can be repaired using all-metal, porcelain-fused-to-metal (PFM), or ceramic restorations. All-metal restorations have undesirable aesthetic appearances and have been criticised by their potential risk of allergic reactions, while PFM fixed partial dentures (FPDs) require much teeth preparation, with a reduction of a minimum 1.5 mm in thickness for both strength and aesthetic reasons [1,2].

During the last decade high-strength zirconia has been increasingly applied in dentistry to replace metal as a coping material in PFMs, wherein a porcelain layer is still required to mask the white colour of zirconia and to generate acceptable aesthetics. It has, however, been reported that chipping and/or delamination of the veneer layer has become a primary complication of the veneered zirconia restorations [3,4]. The reported failures rate of the veneered zirconia restorations in clinical evaluations was up to 30% after 2–5 years [5]. This has driven the development of monolithic zirconia crowns and bridges with full anatomic contour, i.e. monolithic zirconia restorations without the need of adding a veneer layer. Along with the improvements in the colour and translucency of zirconia materials, and with the development of the net-shape fabrication techniques base on computer-aided design and computer-aided manufacturing (CAD/CAM) principles [6], several grades of full-contour zirconia restorations are available nowadays among them the latest one is known as the self-glazed zirconia (SG) with superior surface smoothness that mimics the optical appearances of the enamel of the natural teeth [7].

Yet, the wear and long-term reliability of full-contour zirconia restorations remain two major concerns for dentists and researchers in the field. An 'ideal' restoration material should have good wear-resistance and not induce excessive wear in the opposing natural teeth. Although it was thought that the major risk of full-contour zirconia restorations was that they may induce excessive wear of the opposing natural teeth [8–11], there have been reports revealing that the monolithic zirconia caused less wear of the antagonist enamel than the natural teeth [12–15]. Base on the various results reported so far it can be concluded that the wear of the opposing natural teeth is determined by the surface microstructure and roughness rather than the hardness of the zirconia restoration [2,14,16–22].

According to most results, polishing is an acceptable treatment for optimising the tribology behaviours of the zirconia restorations, which can achieve a smooth surface with low roughness, to protect the opposite natural teeth from excessive wear [17,23]. However, complex manual operation steps involved in the manufacturing process would degrade the dimensional accuracy of the restorations and may induce margin damage and even micro-cracks beside consuming more production time [24,25]. Thus, a material with

a very smooth surface after sintering that does not need polishing would, in practice, be preferable.

The surface roughness of the zirconia restorations is determined mainly by the defects and peeling-off of the particles/grains occurring during the fabrication processes. The commonly used zirconia in dentistry, referred to as 'conventional' zirconia, is made by a multi-step process, starting with the cold isostatic pressing of the 3 mol-% yttria-stabilised tetragonal zirconia polycrystal (3Y-TZP) powder, followed by partial sintering to obtain blanks suitable for dry milling, and a further sintering of the milled products. The inhomogeneous packing of the powder granules may yield the inhomogeneous distribution of density and even the formation of voids in the green bodies that can hardly be healed or removed by partial sintering. During the subsequent milling of pre-sintered or partially sintered zirconia blanks the particles/grains are peeled off one-by-one or in groups depending on the local density, leaving behind a rough surface on the scale of micrometers with deeper ditches where voids are present or where groups of grains are removed at the same time [26].

The novel SG dental restorations are prepared by a precision additive 3D gel deposition approach followed by a milling of the internal surface of the green bodies during which the possible local plastic deformation enable the removal of inter-granule defects thus to minimise the defects concentration and to achieve a smoother milled surface. The application of a wet-chemistry process based on additive manufacturing principle also allows the formation of green bodies with gradient structure and much more improved homogeneity that can be sintered at reduced temperature to achieve fine-grained microstructure besides the formation of a smoother surface [27]. Thus, the new grade of zirconia restorations can inherently form an enamel-like surface, meaning it is very smooth on a micrometer scale, yet with nanoscale roughness that has been approved to have almost the same friction and wear performance against tooth enamel as a well-polished zirconia surface [26].

The aim of this work is to evaluate the long-term reliability of this new grade of zirconia restorations in comparison with their conventional counter parts. Thus, the effect of fatigue on the fracture strength and the fracture mode was assessed.

Material and methods

Sample preparation

The mandibular second premolar and second molar served as abutments for the three-unit mandibular FPDs. The abutment teeth were prepared with a 0.5-mm chamfer width and 5-mm height in a mandibular model. A 3D laser system (Imetric, IScan D104i, Imetric 3D SA, CH) was used to scan the model for the CAD software (Siemens 840D solutionline, Siemens, Germany) to design a master model (selecting the range from 35 to 37 in working dies). The selected range was then milled with a CAM system (DMG MORI, Ultrasonic 10, DMG MORI Seiki, China) using titanium alloy (XiJing, TA1, ShanXiXiJing, China) as a master model in which the bottom face was vertical to the long axis of the abutments (Figure 1). The distance between the centres of the abutment was 13.3 mm. The FPDs were designed with 0.5 mm minimal occlusal thickness, 0.8–1.3 mm axial thickness, about 10 mm pontic width, and a 20 mm^2 connector area using a CAD system.

Two groups of zirconia FPDs were prepared: Group 1 (SG): self-glazed zirconia and Group 2 (CZ): conventional zirconia. The SG restorations were formed by a precision additive 3D gel deposition approach followed by a milling of the internal surface of the green bodies, so the restorations have a smooth self-glazed occlusal surface opposite to a hierarchically rough interior surface. The CZ restorations were shaped by milling the partially sintered blanks, yielding rough surfaces both interior and on the occlusal of the restorations. Both groups of FPDs were sintered to full density at 1450°C for 2 h in air to achieve a relative density above 99.9%. After that, the specimens were furnace cooled down to the room temperature. The SG is a product under development with the commercialisation potential thus more processing details cannot be disclosed here due to the conflict of interest.
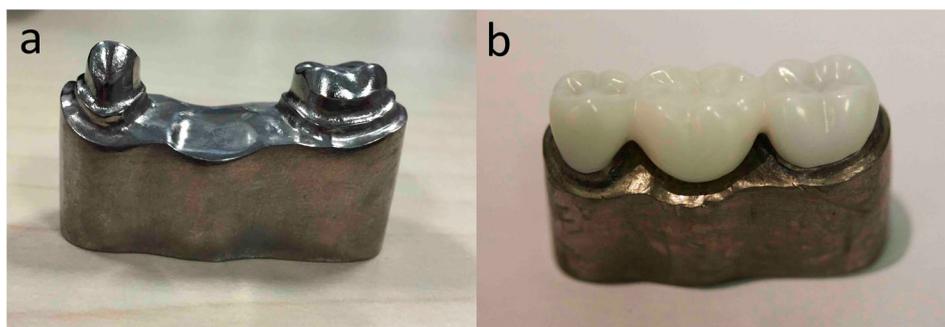

Figure 1. (a) Master model of FPD made of titanium alloy; (b) a SG FPD on the master model.

In total, 32 specimens were prepared for SG (SG) and CZ FPDs ($n = 16$/group) to replace the lower first molars. Each test group was divided randomly into two subgroups ($n = 8$ each): one subgroup was subjected to fatigue testing (F, SG-F, and CZ-F) while specimens in the other subgroup were not fatigue-tested prior to the fracture test as control groups (C, SG-C, and CZ-C). For CZ FPDs, each was polished for 15 min, from rough to fine (Toboom RD-2; Toboom Dental Co, Ltd, Shanghai, China), to simulate the normal clinical process.

Measurement of the grain size and the observation of the post-treated surfaces

Specimens for each group were cut in the vertical plane (Buehler, Isomet 5000, Buehler Ltd, U.S.A.) to expose the cross section, which were then polished (Buehler, Ecomet 250) and subsequently thermal etched (annealing at 1200°C for 1 h in air) and coated with gold for scanning electron microscopy (SEM) observation to determine the grain size and the content and size of defects, if any.

Fatigue test

Each FDP (SG and CZ groups) was cemented onto master model abutments with resin adhesive (Kerr, Maxcem Elite Chroma, Kavo Kerr Dental Co., U.S.A.) and cured with light (Coltent Coltolux LED, C7970, Coltene Whaledent Inc., U.S.A.). Then, eight FDPs from the SG-F and CZ-F experimental groups were subjected to cyclic loading in a fatigue machine (Instron, ElectroPuls E3000, Instron Corp., U.S.A.) for 5 000 000 cycles under a load varying between 30 and 300 N with a frequency of 15 Hz. Loading was applied through a 2-mm diameter stainless steel ball placed perpendicularly at the central fossa of the pontic to simulate the antagonistic surface of the opposing tooth. To avoid high stress concentration in the material, a 0.5-mm piece of ethylene vinyl acetate foil was placed between the ball and the occlusal surface of the pontic. A cyclic loading between 30 and 300 N was selected to simulate the masticatory force that may lead to subcritical crack growth, meaning that the zirconia sample is subjected to a long period of intermittent stresses below the critical stress level ($<K_{Ic}$) but above a threshold stress level ($\geq K_{I0}$) under which the pre-existing flaws may grow slowly in areas of stress concentration and resulting in failure of restoration. The selected load in this range is similar with those reported in early works, i.e. in the range of 0–300 N for the 3-unit FDPs [28].

Fracture test

The non-fatigue-tested specimens and fatigue-tested specimens that did not fracture during fatigue test were mounted individually in a universal testing machine (Instron 5969, Instron Corp., U.S.A.) and loaded to fracture at a 2 mm min^{-1} crosshead speed. This load was applied at the same position on the pontic as in the fatigue test until the fracture of the restorations; the fracture force recorded represents the minimum force to initiate the fracture of the restoration.

Fracture origin determination

After the fracture test, pieces of fractured specimens from each group were coated with gold for SEM observation (FEI, Quanta 200F, FEI Corporation, U.S.A.) to assess the fracture features in selected areas.

Phase analysis

An X-ray diffractometer (Rigaku, SmartLab, Rigaku Corp., Japan) was used to quantitatively analyse the phase transformation of zirconia occurring on the fracture surface. The XRD patterns were recorded by using CuKα radiation (wavelength of 1.5416 Å) over a 2θ range of 20–40° at a step interval of 1 s and step size of 0.03°.

Statistical analysis

Fracture forces were analysed statistically using independent-sample T-tests with the SPSS software (ver. 13.0). Statistical significance was set at $\alpha = 0.05$.

Results

A fewer of voids were observed in the CZ restorations, while its presence was negligible in the SG restorations (Figure 2). Generally, the grain size in the SG restorations was about 200 nm that is sufficiently smaller than that observed in CZ counterparts being 350–600 nm for (Figure 2). No specimen showed fracture or decementation during the fatigue test. Figure 3 shows the fracture force of both materials with and without subjecting to the fatigue test prior to the fracture test. The fracture force of the SG restorations was significantly higher than that of the CZ restorations, both under the conditions with and without subjecting to the fatigue test prior to the fracture test. For both materials, the fatigue test induced a slight increase in fracture force, but the difference was not significant. Figure 4 shows the typical fractographic features observed on the fracture surface. In the case of self-glaze zirconia restorations the fracture always originated at the tip of the connectors where the maximum tensile stress was expected, whereas in the case of CZ restorations the fracture was observed always slightly above the tip of the connectors, i.e. it braked before the maximum tensile stress was applied to the tip of
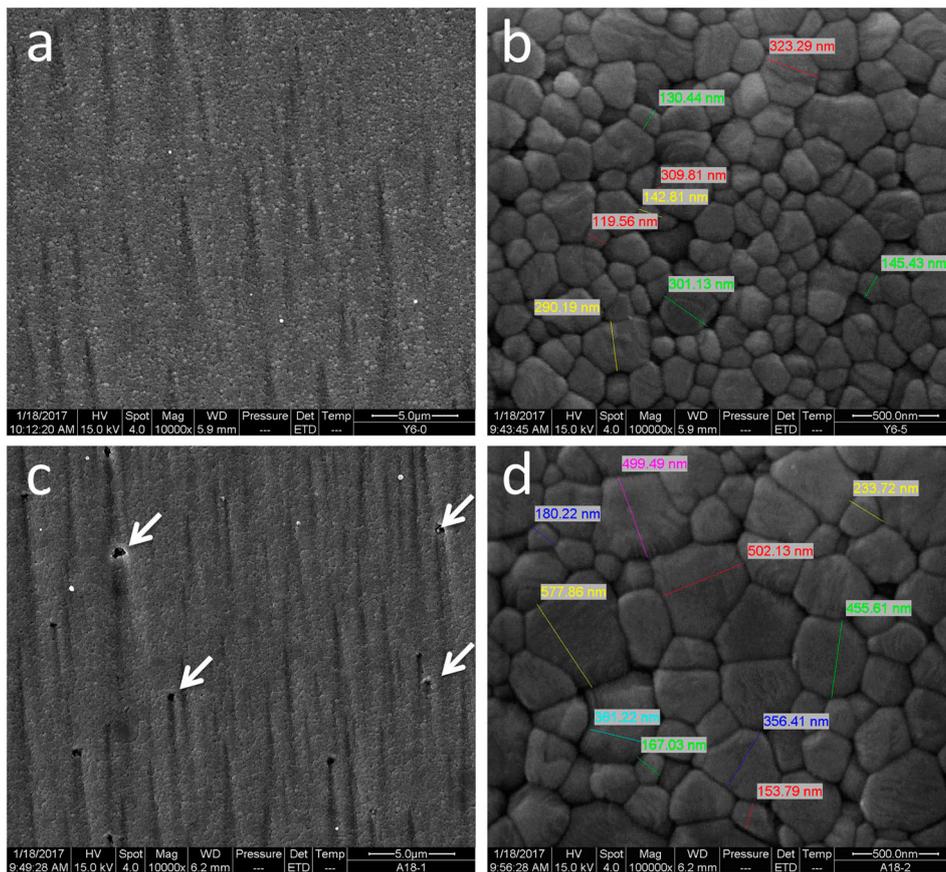
Figure 2. (a) The polished cross section of SG showing relative smooth surface; (b) the polished and thermal etched surface of SG revealing the size and distribution of grains under the magnification of ×100 000; (c) the polished cross section of CZ revealing the presence of voids indicated by the arrows; (d) the polished and thermal etched surface of CZ revealing the size and distribution of the grains under a magnification of ×100 000.

the connectors. There were no obvious voids or any other types of microscopic defects observed at the fracture spot of the SG restorations (Figure 4(a,b)), under high magnification the fracture origin can be identified as surface ditches. In contrast, voids were observed at the fracture spot of the CZ restorations

(Figure 4(c,d)), under high magnification the fracture origin can be identified as a voids-containing low density volume. As shown in Figure 5, the as prepared materials were consisted of monophasic tetragonal zirconia. Without subjecting to fatigue very small amount of m-phase (4.2 and 5.6% for the specimens SG-F and CZ-F, respectively) was observed on the fracture surface in both groups of samples, whereas after subjecting to fatigue increasing amount of m-phase was observed on the facture surface of both groups of materials (Figure 5).

Discussion

Both materials performed well under 5 000 000 cycles of fatigue, simulating 20 years wear in clinical service [29]. In the *in vitro* tests, none of the FPDs fractured, indicating that both the SG and the CZ restorations would meet the clinical requirement in terms of mechanical concerns.

Dry pressing is a common method for fabricating zirconia blanks, which inevitably introduces microscopic voids because of the less homogeneous packing of the powder granuless [26]. Such voids can hardly be removed or healed in subsequent manufacturing processes. Instead, during the sintering procedure, such
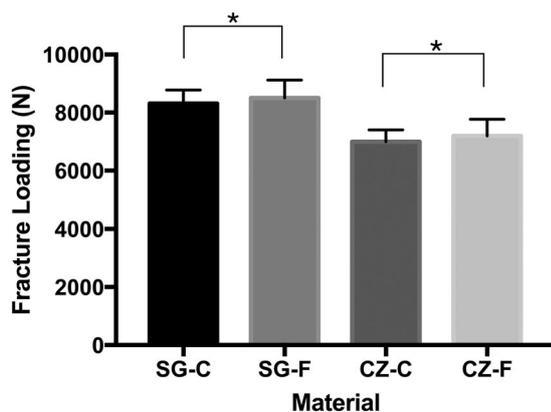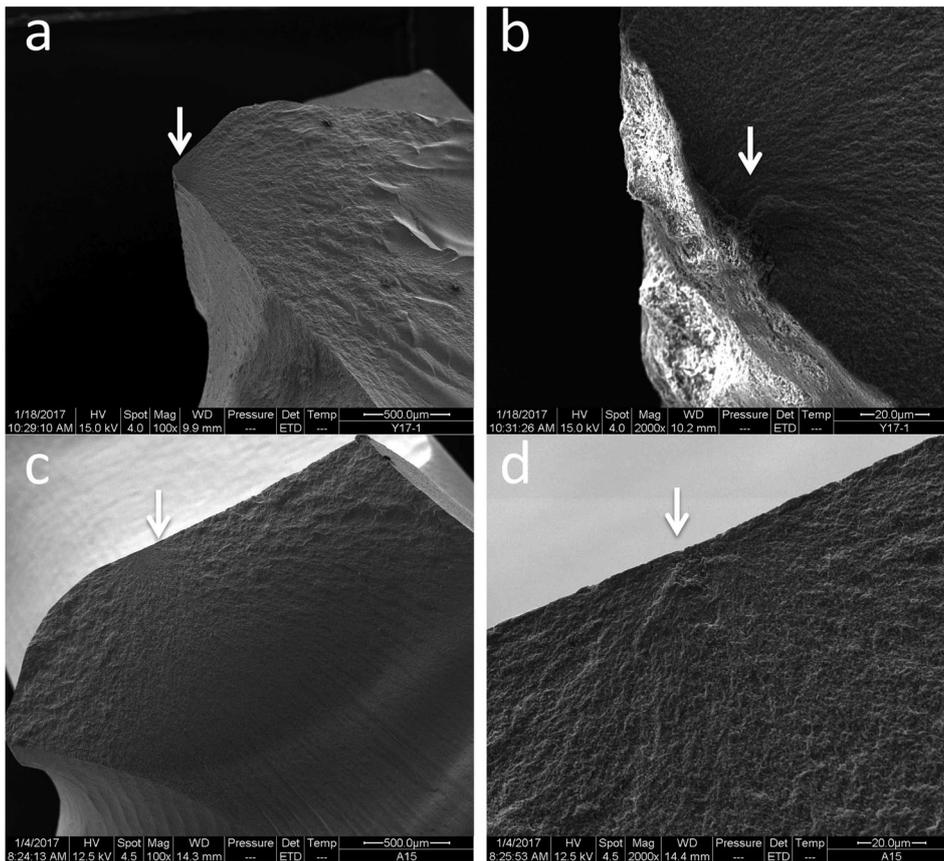

Figure 3. The recorded fracture loads with and without subjecting to fatigue. SG-C: self-glazed zirconia for non-fatigue test; SG-F: self-glazed zirconia for fatigue test; CZ-C: conventional zirconia for non-fatigue test; CZ-F: conventional zirconia for fatigue test. *Fracture loading has no statistical significant difference.

Figure 4. (a and b) Fracture origin of SG; (c and d) fracture origin of CZ.

microscopic voids may grow larger by merging with adjacent pores [30], and in the milling process, these voids will be exposed and material may break off along the large voids, forming new chipping and even cracks. Additionally, the size and location of the defects formed during fabrication processes can affect the lifetime of the restorations. The present study revealed the evident presence of more inherent microscopic defects in the CZ specimens (Figure 2), where the big voids (indicated by the white arrow) may act as trigger for fracturing leading to faster crack propagation [31,32] at relatively low stress, and may induce microstructural degradation [32–34] under long-term or repeated tension-compression stress. The location of a defect, in a tension site or connector area, could also lead to failure at a lower stress [31]. According to the reported clinical failure cases and the most *in vitro* experiments, the fracture would initiate mostly in the basal area of connector or pontic of the three-unit mandibular FPDs under tension and propagated through the pontic occlusal surface, near the contact zone which suffered damages from loading force [35,36].Within the limitations of this study, microscopic defects presented at tension surfaces or connector areas were identified as the fracture origins for CZ restorations that contained obvious fabrication defects (Figure 2(c)). Caution thus should be taken that when such microscopic defects are exposed to the surface, water in the atmosphere may contribute to the easy growth of micro-cracks around

the intrinsic defects under sustained loading known as stress corrosion cracking [1–5,32,37,38]. Such defects and the subsequently induced weakening mechanism will accumulate, and ultimately be responsible for the reduction of the lifetime of the restorations [39]. In contrast, with the precision additive 3D gel deposition process the initial nanoparticles were packed with high homogeneity and density avoiding the inclusion of intergranular voids with higher coordination numbers [26,27]. The SEM images taken on the cross-sectional surface of the sintered bodies confirmed the presence of packing voids in the CZ but not in the SG restorations (Figure 2). Under even high magnification, the SEM images taken on the cross-sectional surface of the milled bodies before sintering revealed the more homogeneous packing of initial particles with higher packing density in SG than that in CZ restorations (Figure 6). In the latter case aggregated secondly particles are clearly visible that would deteriorate the local densification homogeneity.

According to FPDs geometry and the position of loading, the stress distribution observed was similar to a three-point flexure bar. Thus, the maximum tensile stress was located on basal surface of the pontic, and especially the applied load is on the centre of pontic, the maximum tensile stress are higher and located on basal surface of connector. Previous studies have shown that the maximum local tensile stress (MPa) is higher than compression stress (MPa) from applied
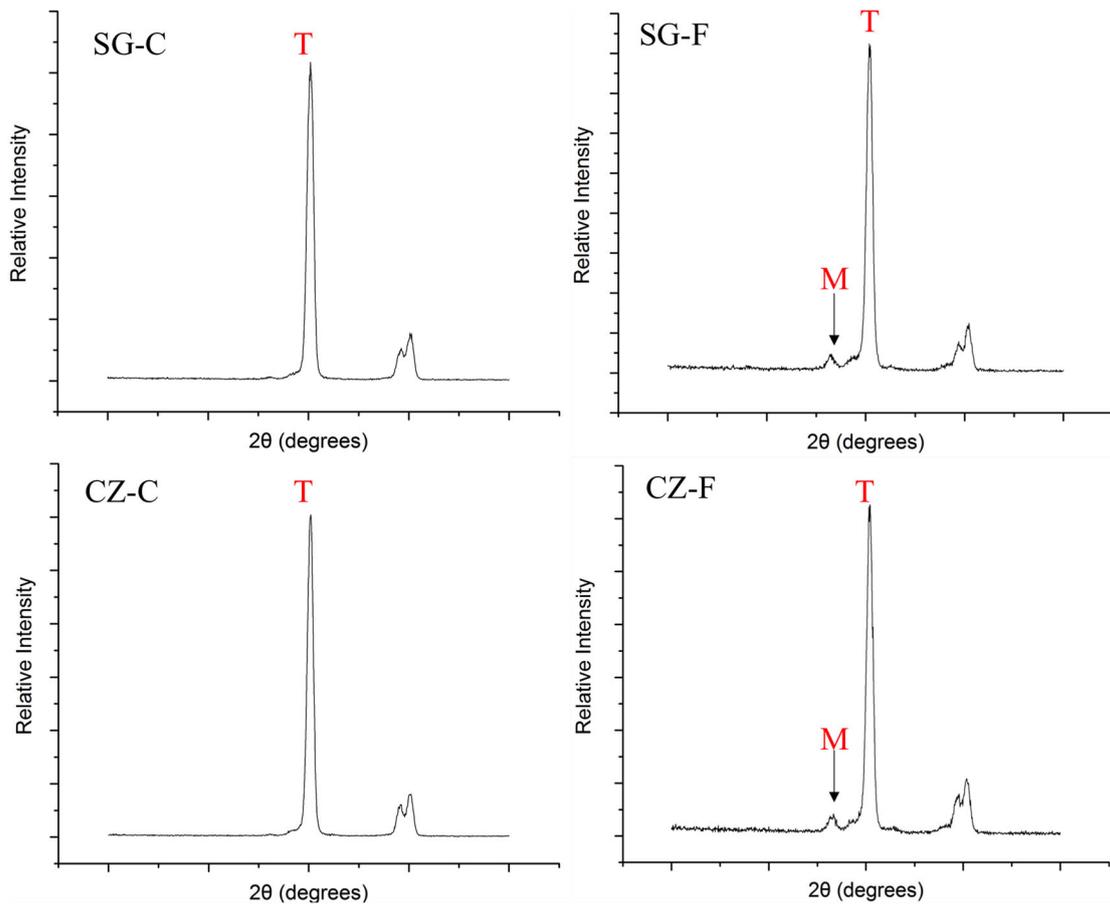
Figure 5. Monoclinic phase was observed only on the fracture surface of both the SG and CZ specimens subjected to cycling fatigue test.

load [40,41]. For sake of comparison it is worth to mention that the measured bending strength of the SG is 1120 ± 70 MPa [7].

The reduction of grain size would decrease the phase transformation (t→m) ability, meaning that the more fine-grained the material is, the less monoclinic zirconia would form during fracture [42–44]. This is because decreasing the size of the zirconia grains can lower the thermodynamic driving force of the transformation, thus to enhance the stability of the tetragonal phase [45–47]. In the present case, very small amount of m-phase was observed on the fracture

surface of the restorations made of both materials without subjecting to the fatigue test prior to the fracture test, indicating that the commonly quoted phase transformation toughening mechanism did not contribute to strengthening the materials. The fact that increased amount of m-phase was observed on the fracture surface of the restorations made of both materials subjected to the fatigue test indicated that the applied cyclic fatigue prior to the fracture test reduced the stability of t-phase. This may explain the observed evidence that the groups of specimens subjected to fatigue test prior to the fracture test demonstrated even slightly
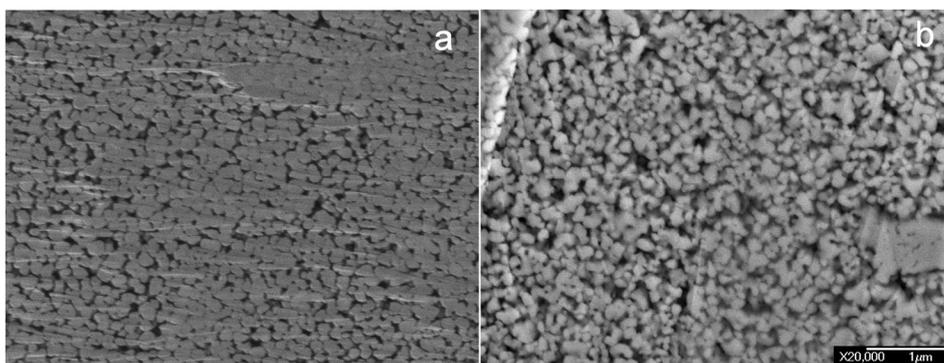

Figure 6. Backscattered SEM micrographs taken on the polished cross section of the green bodies after burning-off organic binder. (a) SG restorations; (b) CZ restorations.

high strength. Under cyclic fatigue stress, tetragonal phase (t) transforms into monoclinic (m) with 3–4% volume expansion, which would induce compressive stresses at the tip of a crack or surface of the materials, preventing further crack propagation thus contributing to strengthening and toughening [48,49]. Previous studies have analysed the stress distribution in posterior FDPs and stated that the highest maximum principal stress (MPSs) occurred around the occlusal contact point of restoration. However, these stress peaks could be ruled out as being causative for the failures observed. Apart from the stress peaks around the occlusal contact points, the highest MPSs (tensile) within the whole structure were observed in the basal region of the connector of the FDPs [50].

In summary, within the limitations of this study no specimen fractured during cyclic loading. Subjecting to 5 million cycle fatigue, simulating about 20 years in clinical service, did not induce any adverse effects in either materials – self-glazed or CZ. Although all tested restorations had the potential to withstand occlusal forces applied in the posterior region, the SG could more suitably be applied in the anterior regions to satisfy the aesthetic demand by reducing the thickness of the restorations made of a stronger material with improved optical translucency. Both materials showed slightly increased fracture force after fatigue test owing to the stability reduction of the tetragonal phase by fatigue stress. The observed significantly higher fracture force of the SG over the CZ can be attributed to the fine-grained microstructure with no visible microscopic void achieved in this category of new restorations.

Acknowledgements

Disclosure statement

Funding

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