Evaluation of a High Fracture Toughness Composite Ceramic for Dental Applications

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Abstract

Purpose: The introduction of yttrium partially stabilized zirconia polycrystals (Y-TZP) has pushed the application limits of all-ceramic restorations. The mechanical properties of these materials can be further improved by the addition of a secondary dopant phase. The aim of this work was to evaluate the properties of a new nano-composite ceramic used as a dental framework material.

Materials and Methods: The properties of a new ceria-stabilized tetragonal zirconia polycrystal co-doped with alumina (Ce-TZP-Al) were investigated. Y-TZP was used as control. Sixty bars ($20 \times 2.5 \times 1.5$ mm^3) from each material were prepared by cutting CAD/CAM milling blocks. Twenty specimens were used to measure the 4-point flexural strength and the modulus of elasticity of the tested materials. The remaining specimens were used to measure the fracture toughness using indentation strength (IS), single edge notched beam (SENB), and fractography (FR). The thermal expansion coefficient (TEC) was measured using temperature expansion diagrams. The bond strength of the two framework materials to two esthetic veneer ceramics was tested using the microtensile bond strength test (MTBS). Finally, scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX) were used to analyze the internal structure of the materials. One- and two-way analysis of variance (ANOVA) and Bonferroni post hoc tests were used to analyze the data ($\alpha = 0.5$).

Results: The flexural strength and modulus of elasticity of Ce-TZP-Al (856 MPa, 170 GPa) were significantly weaker ($p < 0.001$) than those of Y-TZP (1003 MPa, 215 GPa). The (IS) fracture toughness of the former (19.02 MPa m$^{1/2}$) was significantly higher ($p < 0.001$) than SENB (12.6 MPa m$^{1/2}$) or FR (12.8 MPa m$^{1/2}$) values. These values were significantly higher ($p < 0.001$) than the fracture toughness of Y-TZP (7.4 MPa m$^{1/2}$), which showed statistically similar values using the same three techniques. The measured TEC for the two materials was relatively similar, 10.1 μm/°C and 10.4 μm/°C, respectively. Regarding MTBS values, Ce-TZP-Al had significantly lower bond strength values ($p < 0.001$) and a higher percentage of interfacial failure than Y-TZP, which failed completely cohesively with the two used veneer ceramics. SEM analysis revealed zirconia grains pull out and structural defects at the core–veneer interface for Ce-TZP-Al material, which explained its weak bond to the two used veneers.

Conclusion: Despite the promising mechanical properties of Ce-TZP-Al nano-composite ceramic, its very low bond strength to esthetic veneers leaves such layered restorations highly susceptible to delamination and chipping under function. Further studies are needed to enhance the surface stability of this high fracture toughness ceramic.

The introduction of yttrium partially stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics to the dental field allowed the fabrication of long span all-ceramic fixed partial dentures (FPDs) with increased confidence and greater success rates. The unique properties of these materials, combined with current state-of-the-art CAD/CAM technology, allows for the fabrication of complex and large restorations with high accuracy and precision.[1]

One of the remarkable features of Y-TZP is the unique tetragonal-monoclinic crystal phase transformation that increases the fracture toughness of these materials. In response to mechanical stresses, the crystals located at the crack tip undergo phase transformation, which is accompanied by volumetric expansion, causing a compression field that stops the propagating crack. Consuming the energy of propagating cracks by mechanisms such as transformation toughening, crack shielding, and branching does not cause healing of the already existing crack, but prevents catastrophic crack propagation, resulting in an increased fracture toughness of these materials.[2]

Several factors play a significant role in controlling the fracture mechanics of TZP materials, including the grain size and shape, the type and percent of the stabilizing elements, the size and the distribution of internal flaws, and the presence of prestresses. Yttrium dioxide is the common stabilizing element for the zirconia-based ceramics used in the dental field. One of the disadvantages of Y-TZP is its sensitivity to thermal aging.[3,4] As the Y-TZP framework is commonly subjected to this procedure during baking of the ceramic veneer, through multiple firing cycles, depletion of yttrium from the grain boundaries has been observed. This results in the formation of cubic grains, which affect the structural integrity of the material.[5] Besides yttrium, other stabilizers, such as ceria, magnesium, titanium, and calcium, have also been investigated.[6,7]

Two common approaches have been used to enhance the fracture toughness and mechanical properties of zirconia framework materials. One common technique is to reduce the size of the components of the material to a nano-scale to enhance their properties and improve their performance. Dental zirconia has sub-micron grain sizes, which are optimal for enhancing the fracture toughness of these materials.[8] Another successful technique used to control the properties of zirconia-based materials is the addition of one or more secondary elements, also known as the doping phases.[9,10] This secondary phase changes the internal structure of the original material and alters its mechanical and physical properties. Furthermore, alumina is one of the common dopant materials used to increase both the fracture strength and the fracture toughness of the zirconia-based materials.[11] As the percent of the dopant phase may exceed that of the original material, the term "composite ceramic" has been used to describe the complex internal structure of these hybrid materials.[11]

The aim of this study was to evaluate a new composite ceramic material—ceria-stabilized tetragonal zirconia polycrystal co-doped with alumina. The flexural strength, fracture toughness, thermal dimensional behavior, and bond strength to different veneer ceramics were investigated. The internal structure of the material was analyzed using scanning electron microscopy (SEM), while the basic chemical structure was examined using energy dispersive X-ray microanalysis (EDX). Y-TZP was used as a control.

Materials and methods

CAD/CAM milling blocks composed of ceria-stabilized tetragonal zirconia polycrystal co-doped with alumina (Ce-TZP-Al) were used to prepare the required specimens as discussed further on. Commercially available Y-TZP was used as control. Material properties are summarized in Table 1.

Flexural strength test

Twenty bar-shaped specimens (25 × 2.5 × 1.5 mm^3) of each material were prepared by cutting CAD/CAM milling blocks using a diamond-coated disc under water cooling (Isomet 1000; Buehler, Lake Bluff, IL). The bars' tensile surface was polished in a rotating metallographic polishing device using ascending grits of silicon carbide paper under water cooling and a fixed load of 500 g (Ecomet; Buehler Ltd., Evanston, IL). The bars were placed in a 4-point bending attachment unit (20-mm outer span × 10-mm inner span) and were loaded until failure at a crosshead speed of 0.5 mm/min in a universal testing machine (Instron 6022; Instron Limited, High Wycombe, UK). The load cell was calibrated using standardized specimens, and the crosshead speed was monitored using a computer-controlled vertical displacement program. The flexure strain of the bars was measured using vertically displaced digital micrometers (Millitron; Feinpruf Perthen GmbH, Gottingen, Germany). The maximum load at failure was extracted from a computer-generated file and the flexural strength was calculated. The modulus of elasticity was calculated using the flexural strength and the measured strain. Relevant formulas are mentioned elsewhere.[12]

Fracture toughness test

Forty specimens were prepared from each material as described above. For half the specimens, a Vickers micro-indentation was placed on the center of the polished surface using an indentation load of 19.6 or 196 N. The specimens were loaded in the same 4-point bending set-up described above.

Table 1 Material properties

Material	Manufacturer	Composition (wt%)	Flexural strength (MPa)	Fracture toughness (MPa m$^{1/2}$)	Thermal expansion coefficient (μm/°C)	Zirconia grain size (μm)
Ce-TZP-Al	Matsushita Electric Works, Ltd., Osaka, Japan	Zirconia 75.75% 10%mol Ceria Alumina 24.25%	856 ± 97	19.02 ± 1.1 (IS) 12.6 ± 2.4 (SENB) 12.8 ±.59 (FR)	10.1	0.2
Y-TZP	Degudent, GmbH, Hanau-Wolfgang, Germany	Zirconia 93% Y 5.5% Al 1.5%, silica < 1%	1003 ± 132	0.37 ± 0.3 (IS) 77.42 ± 1.3 (SENB) 7.6 ± 48 (FR)	10.4	0.3

Chuntikal's equation was used to estimate the indentation strength (IS) fracture toughness of the tested specimens[13]:

$$K_{IC} = 0.59(E/H)^{1/8} (\sigma_f P^{1/3})^{3/4}$$

where E is the measured Young's modulus; H is the hardness, measured by making five indentations on the broken specimens; σ_f is the measured flexural strength after indentation; and P is the indentation load.

For the other half of the specimens, a single edge notch was cut on the tensile surface of the bars (0.3-mm thick, 300-μm deep) using a diamond-coated disc (Diamond Wafering Blade; No 11-4254, Buehler) under water cooling. The fracture toughness of the single edge notch beams (SENBs) was calculated using the relevant formulas.[14] Fractographic assessment was used to validate the results obtained from the SENB test.[15,16] Eight broken bars were ultrasonically cleaned, gold sputter coated (S150B sputter coater; Edwards, Crawly, UK), and examined under SEM (XL 20; Philips, Eindhoven, The Netherlands) by one qualified operator, and the critical crack size (C_{cr}) was identified and measured according to the following equation:

$$C_{cr} = 0.71(a_{cr}\ b_{cr})^{1/2}$$

where a_{cr} is the crack depth, and b_{cr} is half the crack width. The fracture toughness (FR) was measured using the following equation:

$$K_{IC} = Y\ \sigma_f\ C_{cr}^{1/2}$$

where Y is a geometric parameter calculated from the stress at the crack tip and the geometry of the crack shape. Y was set to 1.24 in this research, ignoring possible effects of any residual prestresses.[16] All specimens were immediately tested following the preparation procedures. All tests were conducted at room temperature (22°C) and under silicon oil to eliminate any possible influence of the air humidity and prestresses, which could result in slow crack growth of the tested materials.

Evaluation of the thermal dimensional behavior

The linear thermal expansion coefficient (TEC) of the tested materials was measured using a precalibrated electrical furnace and a vertical differential push rod thermal dilatometer device as described by Isgro et al.[17] Cylindrical specimens (5-mm diameter by 15-mm length) were placed in the furnace, and the temperature was increased to 525°C. The linear expansion measuring device was set to zero, and the temperature was lowered to room temperature at a rate of 7°C/min enabling determination of the TEC, which was actually the thermal cooling coefficient in this case.[17] Temperature expansion curves were constructed using the digitally recorded data. These data were used as references for selection of a suitable veneering ceramic as discussed further on.

Microtensile bond strength test (MTBS)

Ten disc-shaped specimens (19.5-mm diameter, 3-mm thick) were prepared from each material and were airbone-particle abraded (P-G 400; Hornisch + Rieth, Winterback, Germany) for 10 seconds using 120-μm aluminum oxide particles at 2 bar pressure (S-U-Alustral; Schuler-Dental, Eberhard-Finckh, Germany). One press-on ceramic material (Cercon Ceram Express; Degudent, GmbH, Hanau-Wolfgang, Germany) and one layering veneer ceramic (Nobel Rondo Zirconia; Nobel Biocare AB, Goteborg, Sweden) were selected to veneer the disc-shaped specimens based on their previously established superior bond strength to a zirconia framework material.[18] The core veneer specimens were cut into microbars, and 40 microbars per material were randomly selected (4 microbars/disc/material). The core veneer MTBS was measured according to techniques published elsewhere.[19]

Evaluation of the internal structure and the basic chemical composition

Highly polished sections were thermally etched at 1250°C for 20 minutes (Austromat 3001; Dekema Dental-Keramiköfen GmbH & Co, Freilassing, Germany) to enhance the visibility of grain boundary regions and the internal structure of the tested materials.[20] The sections were examined under high magnification using SEM, and different phase compositions were identified using EDX (EDAX, Inc., Mahwah, NJ).

Statistics

One- and two-way analysis of variance (ANOVA) and Bonferroni post hoc tests were used to analyze the data. Based on the sample size (n = 20/material/test) and the significance level ($\alpha = 0.05$), the statistical test of choice had a statistical power ($1 - \beta = 0.70$) to detect large effect size differences (f = 0.4), which in terms of material properties would be of clinical relevance.[21]

Results

The 4-point flexural strength of Ce-TZP-Al composite ceramic (856 ± 97 MPa) was significantly lower (F = 15.7, $p < 0.001$) than Y-TZP material (1003 ± 132 MPa). Load-strain diagrams revealed some degree of nonlinearity of the former material before the fracture point, as stable crack growth was observed, indicated by flattening of the load curve, while typical elastic behavior was observed for Y-TZP, as the material immediately fractured once the critical stress level was reached (Fig 1). The calculated elastic moduli were 170 GPa and 215 GPa, respectively.

The calculated fracture toughness (IS) values for Ce-TZP-Al were significantly higher (F = 401, $p < 0.001$) than SENB and FR methods: 19.02, 12.6, and 12.84 MPa $m^{1/2}$, respectively. These values were significantly higher (F = 2778, $p < 0.001$) than the values calculated for Y-TZP (7.4 ± 0.32 MPa $m^{1/2}$) using the same techniques. Accurate determination of critical crack dimensions was hindered by the internal structure of these polycrystalline materials (Fig 2).

The TEC for the Ce-TZP-Al was 10.1 μm/°C, closely matching the TEC of Y-TZP material, 10.4 μm/°C. Temperature expansion diagrams demonstrated apparent linearity over the recorded temperature range (20 to 525°C) (Table 1).

The MTBS of Ceram Express (3.7 MPa) and Nobel Rondo (11.3 MPa) to Ce-TZP-Al were significantly weaker (F = 199, $p < 0.001$) than the values recorded with Y-TZP, 32 and 36.7 MPa, respectively. In addition to premature failure during

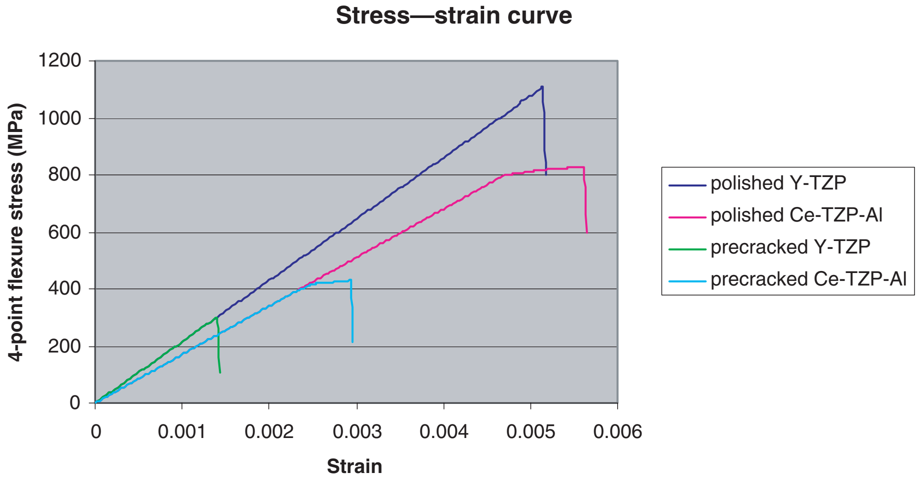

Figure 1 Stress—strain curve of polished and precracked specimens. Y-TZP exhibited a linear brittle behavior as the specimens were fractured as soon as the critical stress level was reached. Ce-TZP-Al specimens demonstrated some degree of plastic behavior, as once the critical stress level was reached, stable crack growth was observed. For the same size of precrack, Ce-TZP-Al specimens were able to sustain higher loads than Y-TZP.

cutting the microbars, spontaneous failure of the microbars during testing resulted in the high standard deviation observed with the Ce-TZP-Al ceramic. SEM examination of the broken Ce-TZP-Al microbars revealed 100% interfacial failure at the core–veneer interface. Additionally, detached zirconia grains were identified on the veneer side of the broken microbars using EDX (Fig 3A). Structural defects between the veneer ceramics and this framework material were also observed at high power magnifications (Fig 3B). On the other hand, Y-TZP microbars failed by 100% cohesive fracture as the crack traversed the two used veneer ceramics (Table 2).

SEM and EDX analysis revealed homogenous grain size (0.3 μm) and internal structure typical of the Y-TZP dental framework materials (TZP 93 wt%, Y 5.5 wt%, Al 1.5 wt%,

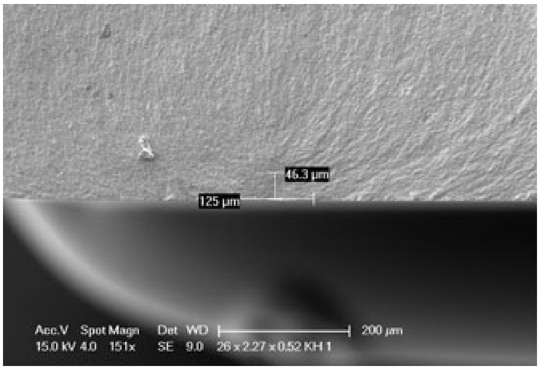

Figure 2 SEM image, 151×, demonstrating a cross-section of the tensile surface of a fractured Ce-TZP-Al bar. Precise measurement of the dimensions of the critical crack (measurement on the image) was complicated by the complex internal structure of the material. Classical crack features of brittle materials such as mirror, mist, and crack branching are hindered by the grain structure of the material.

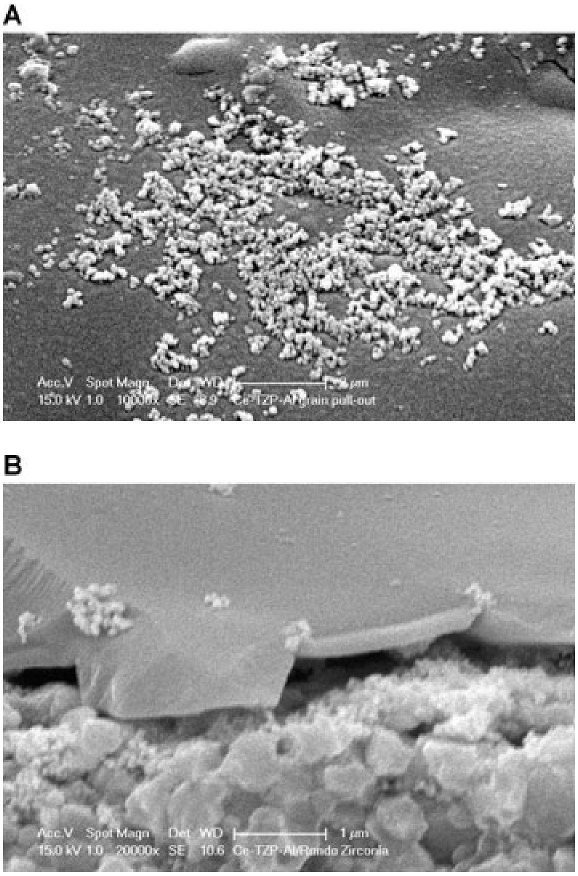

Figure 3 (A) SEM image, 10,000×, demonstrating the veneer side of a broken Ce-TZP-Al microbar showing grain pullout. Grains were chemically identified using EDX. (B) SEM image, 20,000×, demonstrating the core–veneer interface of Ce-TZP-Al specimen showing micro-spaces and zirconia grain pullout.

Table 2 Microtensile bond strength values (MPa) and fracture type for the tested materials

Framework material	Ceramic	MTBS (MPa)	Fracture type
Ce-TZP-Al	Nobel Rondo	11.3 ± 2.6	100% interfacial
	Ceram Express	3.7 ± 3.6	100% interfacial
Y-TZP	Nobel Rondo	36.7 ± 12	100% cohesive
	Ceram Express	32 ± 7	100% cohesive

and traces of silica <1 wt%). The internal structure of Ce-TZP-Al was relatively more complex. On a microscopic level, the material was basically composed of a matrix of Ce-TZP (TZP 75.75 wt% stabilized with 10 mol% CeO_2), which demonstrated some degree of internal porosity compared to the fully dense Y-TZP ceramic. This structure was interrupted by clusters of alumina grains (24.25 wt%) (Fig 4A). On an ultra-structural level, independent nuclei of Ce-TZP were observed inside the alumina grains along with randomly observed nuclei of alumina inside the Ce-TZP islands (Fig 4B, C).

Discussion

The selection of the 4-point bending test for measuring the flexural strength and the fracture toughness offered a more controlled environment for evaluating the mechanical properties of the tested ceramics, but it was more tedious than other test methods, such as biaxial or 3-point bending flexural strength tests, as it requires careful attention, especially during positioning of the specimens in the attachment unit.[22,23]

While the flexure strength of the nano-composite ceramic was significantly lower (856 MPa) than in the Y-TZP material (1003 MPa), its fracture toughness was, on the other hand, significantly higher, 19 MPa $m^{1/2}$ (IS) and 12.6 MPa $m^{1/2}$/12.8 MPa $m^{1/2}$ (SENB/FR) compared to 7.5 MPa $m^{1/2}$ (IS, SENB, and FR) measured for Y-TZP framework.[7,24] A point worth considering is that the fracture toughness of the underlying framework material is a direct measure of its resistance to crack growth and propagation, which reflects higher tolerance toward surface and structural damage. The CAD/CAM milling procedure plus different laboratory surface treatments, such as grinding or airborne-particle abrasion, all induce a certain degree of surface damage and roughness, which could act as crack initiation sites. Thus, a framework with a higher toughness is expected to resist crack initiation, as it would require higher failure loads compared to another material, which has the same surface damage but lower toughness. On the other hand, a framework material with a high modulus of elasticity and low toughness (a brittle framework, such as Y-TZP) would immediately fracture at the first sign of overloading (Fig 1). The flexural strength and the fracture toughness of some contemporary all-ceramic materials are summarized in Table 3.

The calculated toughness using SENB and FR for the composite ceramic used in this study was in accordance with De Aza et al, who studied the effect of the percentage of alumina on the crack threshold value of this composite ceramic.[25] On the other hand, the higher K_{ic} value calculated using the IS method for Ce-TZP-Al compared to SENB or FR values was previously reported by several authors[3,4,26] who also found higher fracture toughness using the IS method. Such significant differences could be related to the nonhomogenous structure of the material, thus the made indentations were placed at theoretically

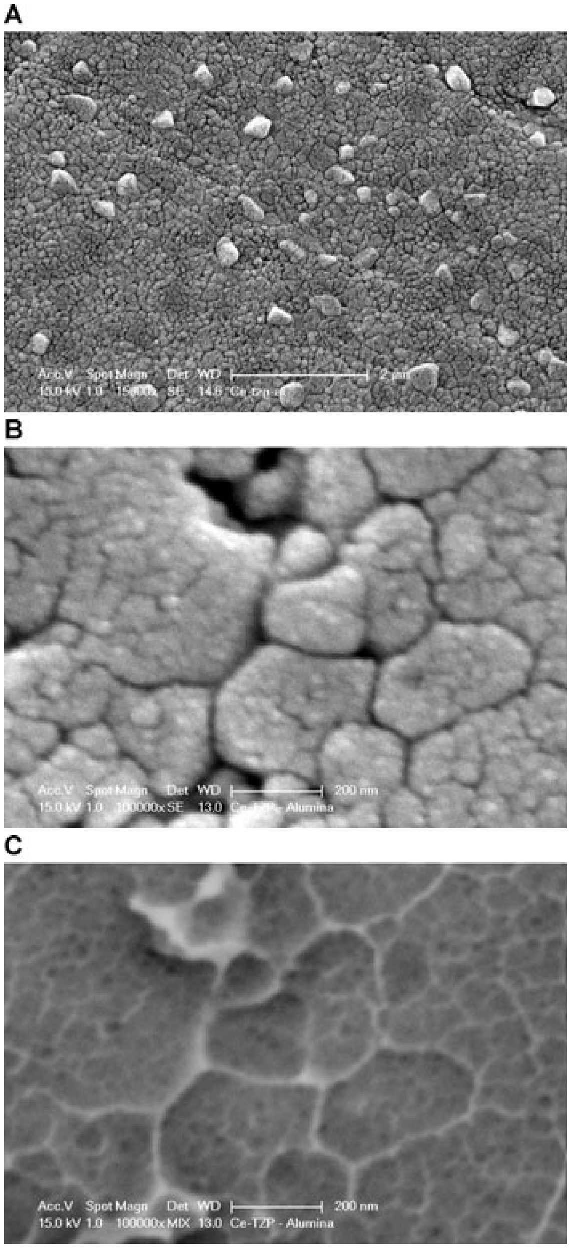

Figure 4 (A) SEM image (15,000×) demonstrating the microscopic structure of Ce-TZP-Al composite ceramic. Matrix of Ce-TZP polycrystals are interrupted by larger alumina grains. (B) SEM image (100,000×) demonstrating the complex internal structure of the nano-composite ceramic. Zirconia grains contain one or more alumina nuclei, which appear as white spots. (C) Inverted contrast of the SEM image in Figure 4B, demonstrating zirconia grain boundaries appearing in white and the alumina nuclei appearing as black spots.

Table 3 Flexural strength (MPa) and fracture toughness (MPa $m^{1/2}$) of all-ceramic materials based on manufacturer data and literature review

	Alumina-toughened zirconia	Zirconia	Alumina	Lithium disilicate	Glass-infiltrated ceramics	Leucite-reinforced ceramics	Glass veneering ceramics
Commercial names	Experimental Ce-TZP-Al	Lava, Cercon, Procera zirconia, ZirCAD	Procera Allceram	IPS Empress 2	In-Ceram	IPS Empress I, Cerec, Vitadur core	Eris, Lava Ceram, Rondo, Ceram Kiss, IPS e.max
Flexural strength (MPa)	700-941	900-1250	400	300-440	320-352	65-134	55-120
Fracture toughness (MPa $m^{1/2}$)	12-20.5	4.2-10.5	4.1-5	3.1-3.7	4.1-4.49	0.9-1.39	0.7-1.2

different phases of the material, and thus the resultant precrack was not standardized. In contrast to other studies recommending IS technique, this method does not account for the presence of structural defects, presence of prestresses between the different phases, and the bond strength between these phases, which could explain the recorded higher toughness using IS method.[27]

Additionally, linear reduction in flexural strength and fracture toughness values was previously proven to be directly related to the percentage of structural defects and pores present in a homogenous single-phase test material.[28] On the other hand, Ce-TZP-Al is a multi-phase material where flexural strength was inversely affected by the presence of structural defects; nevertheless, its structural complexity and material properties resulted in increasing its fracture toughness, as a propagating crack would have to pass through the different phases of the material and to travel in a tortuous path following the different sizes of the grains, all of which result in hindering crack growth and in dissipation of its energy.

It was also previously reported that variations in the toughness of ceria-stabilized zirconia are related to the chemical condition of the stabilizing agent and its valency number, which could also explain the observed discrepancy in fracture toughness values.[29]

The estimated fracture toughness of Y-TZP was consistent with results of other investigations where the same techniques and similar materials were used.[1,30] The nonsignificant minor differences between IS, SENB, or FR values were related to marginal errors made during measuring critical crack dimensions of the tested specimens.[31]

The previous mechanical properties were directly related to the internal structure of the tested materials. Y-TZP was composed of densely packed single-type homogenous sub-micron grains (0.3 to 0.5 μm) without any observed structural defects or flaws. While this structure resulted in a high flexural strength value, it offered less resistance to crack propagation, which, once initiated, will travel at very high speeds, giving no sufficient time for the tetragonal-monoclinic transformation toughening mechanism to interrupt the already advanced crack tip. Monoclinic phase was not detected in crystallographic examination of broken Y-TZP specimens, which supports this opinion.[32] Decreasing the percentage of yttrium might improve the fracture toughness of Y-TZP by enhancing its transformation capacity.[9]

The complex internal structure of Ce-TZP-Al material offered more resistance to crack tip propagation, which was frequently interrupted by the larger alumina grains, causing its deflection and thus dissipating some of its energy. At the same time, it is possible that the neighboring zirconia grains became stress activated to undergo tetragonal-monoclinic transformation ahead of the propagating crack tip. On the other hand, this same complex structure resulted in a higher percentage of internal porosities and defects, which was responsible for its inferior flexural strength, which necessitates paying attention to the processing root of this material.[11] The long-term fatigue behavior of such composite ceramic would be affected by the interaction of these variables.[33]

The linear TEC of both materials was relatively similar, and any dental veneer ceramic with a closely matching TEC could be used for layering these framework materials.[17] Regarding the core veneer bond strength, the reported values with Y-TZP material, 36.7 MPa for Rondo Zirconia and 32.7 MPa for Ceram Express, were slightly lower than previously reported for the same materials.[18,19] A possible explanation is that the zirconia used in the present work was colored, while white zirconia was used in the other studies. A recent study related such findings to the presence of coloring pigments in yellow zirconia frameworks, which weakened the bond strength with different veneer ceramics.[34]

The very low MTBS values observed between Ce-TZP-Al and the two used veneer ceramics was a striking finding. SEM analysis of the broken Ce-TZP-Al microbars revealed zirconia grains pull out, which indicates that these surface grains are not well fused to the surrounding alumina grains and to the remaining structure. Examination of the core–veneer interface for this material demonstrated micro-gaps between the esthetic veneer and the composite ceramic and detached zirconia grains, which corresponds well with the grain pullout phenomena, the low MTBS values, and the premature failure observed for this material (Fig 3A, B).

This behavior could be explained based on the fact that the alumina grains prevent nucleation of zirconia monoclinic phase and prevent transformation propagation to neighboring zirconia grains, and due to the mismatch in the elastic module between alumina and zirconia, the transformation of bulk grains becomes severely restricted. On the other hand, surface grains can accommodate such strain in a vertical direction, which may lead to grain pop-out and detachment.[11]

Atomic force microscopy indicated a difference in the martensitic surface relief between zirconia stabilized with yttrium and those stabilized with ceria. For Y-TZP, self-accommodating variant pairs created a rippled structure on the grain surface, which progressed to involve the whole grain. For

Ce-TZP, these variant pairs were limited to part of the grain surface and accompanied by very high stresses, which may also explain the observed grain pullout of the composite ceramic used in this study.[20]

Conclusion

Based on the findings of this research, the mechanical properties in terms of flexural strength and fracture toughness of Ce-TZP-Al were suitable for short- and intermediate-span FPD restorations. On the other hand, its very low bond strength with the veneer ceramics would make such a layered restoration highly susceptible to chipping and delamination failure under function, which needs further study.[35]

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