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RESEARCH AND EDUCATION

Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia

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ABSTRACT

Statement of problem. Recent monolithic zirconia materials used for indirect restorations are predominantly fully stabilized zirconia with claims of enhanced optical properties. These restorations may behave differently from the conventional partially stabilized zirconia restorations, which may negatively affect some of the core properties required for restoration success.

Purpose. The purpose of this in vitro study was to evaluate and compare the effects of staining, airborne-particle abrasion, and artificial aging on the flexural strength of fully and partially stabilized zirconia material.

Material and methods. Each partially stabilized monolithic zirconia (PSZ) and fully stabilized zirconia (FSZ) material and a zirconia core material (control) were prepared as bar-shaped specimens (2×2×25 mm) and divided into 6 groups (n=8/subgroup): regular sintering, vacuum sintering, stained, airborne-particle abrasion, artificially aged regular sintering, and artificially aged vacuum sintering. Critical load to fracture was determined for all groups by using monotonic uniaxial loading in accordance with International Organization for Standardization standard 6872. Data were analyzed using univariate analysis of variance, followed by the Tukey honest significant difference post hoc test (α=.05).

Results. The control and PSZ (1034 and 1008 MPa) displayed a significantly higher ($P<.05$) flexural strength than FSZ (582 MPa). Airborne-particle abrasion significantly ($P<.05$) enhanced the strength of the control and PSZ (1413 and 1227 MPa) but significantly ($P<.05$) reduced the flexural strength of the FSZ (442 MPa). Staining, artificial aging, and vacuum sintering had no significant effects on any of the groups.

Conclusions. Fully stabilized zirconia may behave differently from conventional PSZ, especially with regard to airborne-particle abrasion, which may weaken the FSZ. The strength of PSZ is approximately double the strength of FSZ. Both of the zirconia materials showed resistance to artificial aging. (J Prosthet Dent 2017;118:216-220)

Mimicking natural teeth has been the great challenge in fabricating monolithic zirconia restorations because of their inherent semitranslucent optical properties.[1] Manufacturers have been striving for a more translucent monolithic zirconia restorative material to fulfill the extremely high demand of the market.

Zirconia is a well-known polymorph that exists in its monoclinic (*m*) form at room temperature and in its tetragonal (*t*) form in temperatures of 1170°C to 2370°C, at which point it transforms into its cubic (*c*) form. Cooling zirconia to room temperature produces volume expansion between 3% and 4% as a result of *t*-to-*m* phase transformation; this in turn generates expansion stresses causing cracks to propagate within the pure zirconia ceramic and shattering it.[2] Stabilizing oxides such as CaO, MgO, CeO_2, and Y_2O_3 have been added to pure zirconia to stabilize it at room temperature.[3] Tetragonal zirconia polycrystals contain between 2% and 3% Y_2O_3 and are composed entirely of tetragonal grains.[4] This form of zirconia was introduced into dentistry in the early 1990s as yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), or, owing to the percentage of Y_2O_3, as PSZ. Tetragonal grains are metastable in nature and can transform

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Clinical Implications

Airborne particle abrasion may weaken fully stabilized zirconia restorations, a procedure that is routinely performed before bonding monolithic zirconia restorations.

spontaneously to the monoclinic form, inducing compressive stresses under the surface and ultimately leading to failure of the material. Mechanical properties of PSZ are highly influenced by grain size and yttria content.[5]

In efforts to improve the optical properties of monolithic zirconia, manufacturers have produced yttria-stabilized zirconia by adding more cubic zirconia and increasing the yttria content anywhere from 6% to 8%, with its stabilization highly dependent on the sintering temperatures.[6,7] Such a form of zirconia, termed FSZ, has been reported to have physical and mechanical properties inferior to those of PSZ and flexural strength as low as 600 MPa.[8]

Surface treatments such as grinding and airborne-particle abrasion of the intaglio of the zirconia restoration have been proposed to improve bonding. This process increases the strength of the Y-TZP provided that the ceramic is not subjected to heat, cyclic loading, or polishing, which can compromise the compressive stress layer and enhance crack propagation.[9,10] Staining or coloring of PSZ decreases the flexural strength of PSZ, with minimal enhancement to its optical properties.[8] The stability of PSZ may be compromised by low-temperature degradation (LTD), which results in a spontaneous transformation of the metastable *t* phase to the *m* phase in the presence of water at a temperature of 200°C.[11,12] Reducing the grain size and/or increasing the concentration of stabilizing oxide reduces the transformation rate,[13] and certain surface treatments may enhance the resistance of Y-TZP to LTD.[14]

As FSZ has been promoted to dentists as a more translucent zirconia restoration, research into the effects of surface treatments on the properties of FSZ is needed. The purpose of this study was to compare the effects of different sintering furnaces, airborne-particle abrasion, staining, and the process of aging on the flexural strength of FSZ versus on that of PSZ.

MATERIAL AND METHODS

Two types of monolithic zirconia material, categorized according to their degree of stability, were used in this study (Table 1): PSZ (Prettau; Zirkonzahn) and FSZ (Prettau Anterior; Zirkonzahn). A zirconia core material (ICE zircon; Zirkonzahn) was used as a control. The zirconia materials in their presintered stage were cut into bar-shaped specimens (Struers Secotom-50; Struers) and then ground to 2.5×2.5×31.2 mm, using silicon carbide abrasive paper (FEPA #1200 and 2400 grit; Struers LaboPol 21; Struers) under constant pressure. The final dimensions (2×2×25 ±0.01 mm) were measured using digital calipers (Absolute Digimatic Caliper; Mitutoyo Corp). Specimens were divided into 6 groups (n=8/subgroup): regular sintering, vacuum sintering, stained, airborne-particle abrasion, artificially aged regular sintering, and artificially aged vacuum sintering.

Table 1. Zirconia materials used

Brand Name	Manufacturer	Composition*
Partially stabilized zirconia		
Prettau zirconia	Zirkonzahn	4%–6% Y_2O_3, <1% Al_2O_3, max. 0.02% SiO_2, max. 0.01% Fe_2O_3, max. 0.04% Na_2O
Fully stabilized zirconia		
Prettau anterior	Zirkonzahn	<12% Y_2O_3, <1% Al_2O_3, max. 0.02% SiO_2, max. 0.02% Fe_2O_3, max.
Control (PSZ)		
ICE Zircon	Zirkonzahn	4%–6% Y_2O_3, <1% Al_2O_3, max. 0.02% SiO_2, max. 0.01% Fe_2O_3, max. 0.04% Na_2O

*Manufacturer's data.

Table 2. Sintering parameters used

Brand Name	Heating/Cooling Rate (°C/min)	Sintering Temperature (°C)
ICE Zirkon	6	1500
PSZ	6	1600
FSZ	5	1450

FSZ, fully stabilized zirconia; PSZ, partially stabilized zirconia. Holding temperature at 2 hours for all groups.

Regular sintering, staining, airborne-particle abrasion, artificially aged regular sintering (Zirkonofen 600/V2; Zirkonzahn), and vacuum sintering and artificially aged vacuum sintering (Zirkonofen 700 Ultra-Vakuum; Zirkonzahn) were performed according to the manufacturers' instructions (Table 2). Specimens were cleaned ultrasonically in distilled water for 10 minutes (Quantrex 90; L&R Ultrasonics Manufacturing) and then air dried for 20 seconds.

In their presintered stage, the FSZ specimens were stained with shade A3.5 Color Liquid Prettau Anterior Aquarell (Zirkonzahn), and PSZ specimens were stained with shade A3.5 Color Liquid Prettau Aquarell (Zirkonzahn). A touchup brush was used to apply 2 coats of the liquid stains. The control specimens were dipped into Color Liquid for ICE zirkon shade A3.5 (Zirkonzahn). The specimens were placed for 20 minutes under an infrared drying lamp (Zirkonlamp 250; Zirkonzahn), according to the manufacturers' instructions.

One side of the specimen (tensile side in the 3-point bend test) was airborne-particle abraded with 50-µm Al_2O_3 particles with the nozzle perpendicular at a distance of 10 mm for 10 seconds and a compressed air

Table 3. Effect of treatments, sintering furnace, staining, airborne-particle abrasion, and artificial aging on mean ±SD flexural strength (MPa) of different types of zirconia material

	Treatment					
Zirconia Type	**Regular Sintering**	**Vacuum Sintering**	**Stained**	**Airborne-Particle Abrasion**	**Artificially Aged Regular Sintering**	**Artificially Aged Vacuum Sintering**
Control zirconia	1034 ±116[a,b]	919 ±117[a]	960 ±157[a,b]	1413 ±177[c]	1170 ±111[b]	1133 ±191[a,b]
PSZ	1008 ±110[a]	1044 ±93[a]	926 ±45[a]	1227 ±194[b]	1048 ±145[a]	970 ±75[a]
FSZ	582 ±52[b]	621 ±62[b]	613 ±66[b]	442 ±21[a]	591 ±47[b]	617 ±132[b]

FSZ, fully stabilized zirconia; PSZ, partially stabilized zirconia. [a,b]Effects of treatments, sintering furnace, staining, airborne-particle abrasion, and artificial aging on mean ±SD flexural strength (MPa) of different types of zirconia material. Different superscript letters indicate different means within same row ($P<.05$).

pressure of 400 kPa (Renfert Basic Classic; Renfert GmbH). Low-temperature aging was carried out in a steam autoclave (CISA 200; Cisa S.p.A.) at 125°C under a pressure of 200 kPa for 8 hours.

Critical load to fracture was determined for all groups by using monotonic uniaxial loading in accordance with International Organization for Standardization standard 6872.[15] Specimens were tested dry at room temperature (22 ±1°C) and at a relative humidity of 70 ±5%, using a universal testing machine (model LRX; Lloyd Instruments) equipped with a 10-kN load cell. The load at a crosshead speed of 0.5 mm/min was applied to the midpoint between the supports by means of a third steel knife edge along a line perpendicular to the long axis of the bar until fracture occurred. Data were recorded by a computer program (Nexygen; Lloyd Instruments Ltd).

The different types of zirconia (ICE zirkon, PSZ, and FSZ) were sputter-coated with gold (Polaron SC 502; Fisons Instruments), and secondary electron scanning electron microscopy images were made (Leo Gemini 1530; Zeiss). Visual examination was used to inspect the grain structure of both groups after sintering, and no quantitative data regarding grain size were extracted.

Statistical analysis was performed using software (IBM SPSS Statistics v22.0; IBM Corp). Data were analyzed using univariate analysis of variance followed by the Tukey honest significant difference post hoc test. Type of zirconia, the sintering furnace, staining, and artificial aging were the independent variables. Uniaxial flexural strength was the dependent variable (α=.05).

RESULTS

The overall flexural strength (MPa) of control zirconia was significantly higher than that of PSZ, and the flexural strength of PSZ was significantly higher than of FSZ ($P<.05$). The sintering furnace, staining, airborne-particle abrasion, and artificial aging had a significant effect on the flexural strength values of the different groups of zirconia (Table 3). Control zirconia flexural strength was significantly reduced by vacuum sintering (919 MPa) ($P<.05$), whereas its flexural strength increased significantly after airborne-particle abrasion and artificial aging with sintering in a regular furnace (1413 and 1170 MPa, respectively) ($P<.05$). Partially stabilized zirconia flexural strength was significantly increased by airborne-particle abrasion (1227 MPa) ($P<.05$). Fully stabilized zirconia flexural strength was significantly reduced by airborne-particle abrasion (442 MPa) ($P<.05$). Overall, airborne-particle abrasion was the only variable that had a significant effect on all types of zirconia, increasing the strength of PSZ and control zirconia but decreasing the strength of FSZ.

Visual inspection of the scanning electron microscopy images (Fig. 1) showed that the PSZ and FSZ grains displayed minor crack-like surfaces measuring approximately 180 nm in length. The grain size of the FSZ was evidently larger than that of the PSZ and ICE zirkon, perhaps because the phase composition of FSZ was different from those of ICE zirkon and PSZ.

DISCUSSION

Based on the findings of this study, a significant difference was found between PSZ and FSZ in how they responded to different treatments. Further investigations, more clinically relevant in vitro studies, and clinical trials will help the clinician gain a deeper understanding of the properties of, effect of certain treatments on, and potential life span of zirconia material.

This investigation, focusing on the sintering of zirconia, agrees with studies that reported the sintering process (temperature and time) should be considered as it directly influences the zirconia's grain size, yttrium segregation, and amount of cubic phase, which in turn reflects zirconia's physical, mechanical, and optical properties.[16] Increasing sintering temperatures increases the grain size of zirconia, which may enhance its physical properties but renders the zirconia more vulnerable to LTD. The 2 zirconia materials investigated in this study had different grain/particle sizes, phases, chemical species, and yttrium distribution and were sintered at different temperatures (following manufacturers' instructions). The PSZ was sintered to temperatures as high as 1600°C, and the FSZ was sintered at 1450°C. The increased flexural strength of PSZ over that of FSZ is in agreement with findings from other studies[8] and supports manufacturers' claims; how that will translate into the long-term success of either type of zirconia material has yet to be established.

Sintering in a vacuum furnace in an attempt to improve the physical and optical properties of zirconia is based on

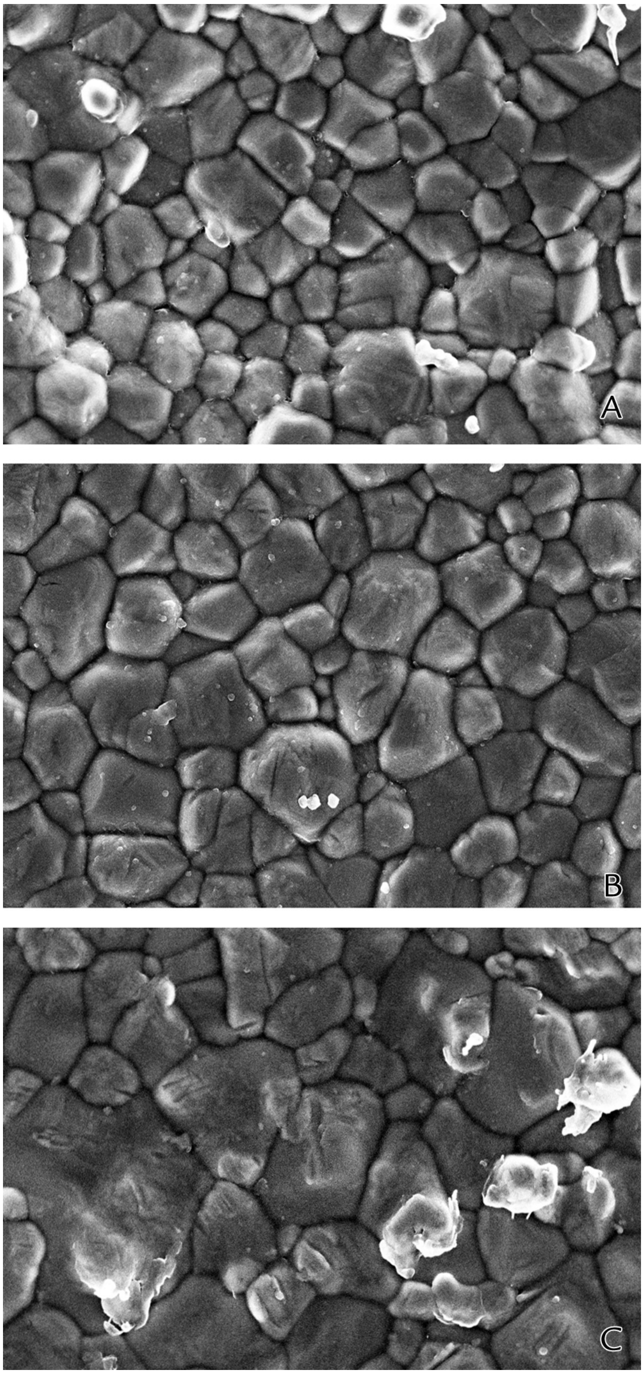

Figure 1. Scanning electron microscopy images after sintering in a regular furnace. A, ICE zircon. B, Partially stabilized zirconia. C, Fully stabilized zirconia (original magnification ×10 000).

the notion that the absence of air and other gases prevents heat transfer within the product through convection and removes a source of contamination, which in turn may affect the chemical purity and metastability of the zirconia material.[8] Based on the results of this study, this concept seems to only minimally enhance the strength (statistically insignificant; $P<.05$) of the FSZ and PSZ compared with sintering in a regular furnace. The strength of the control group was significantly reduced, with no obvious explanation, ruling out any obvious benefits for selecting these higher priced vacuum-based furnaces.

Staining of monolithic zirconia restorations is essential for an esthetic restoration. One of the staining methods relies on the infiltration of solutions containing metal-salts applied to the zirconia material in its presintered stage. These metal-salts interact with the zirconia particles through crystallographic and microstructural changes, which may influence the physical properties of zirconia material.[17] This was also reported by Sulaiman et al,[8] who stated that metal-salts coloring liquids significantly enhanced the strength of FSZ but decreased that of PSZ. One possible theory is that FSZ possesses different microstructure and grain size, higher yttria content, and more intergrain spaces. This may increase the uptake of the coloring liquids, thereby enhancing its physical properties through microstructural changes and yttria preservation.

Airborne-particle abrasion of the intaglio of zirconia restorations significantly enhances its bond strength to the luting agent and has been recommended for clinical use.[18] This abrasive process transforms the impacted tetragonal zirconia particles to their *m* phase particles, inducing protective compressive stresses, preventing crack propagation, thus initially enhancing its flexural strength.[2] However, these stress-generating treatments alter the structural stability of the zirconia particles, which may render the zirconia more susceptible to LTD, to deterioration of the compressive stresses along the cracks promoting its progression, and ultimately to failure of the restoration. The structural differences between FSZ and PSZ, in which FSZ possesses more *c*-phase particles and fewer *t*-phase particles, explains why, in this study, airborne-particle abrasion significantly decreased the flexural strength of FSZ and significantly increased the strength of PSZ. However, this increase in strength is reported under dry monotonic loading, which can be misleading for any clinical recommendations; cyclic loading in water may provide a more clinically relevant outcome.

No definitive recommendations concerning the airborne-particle abrasion of FSZ can be made based on the results of this study, and further investigations should be implemented to provide a more thorough explanation. The airborne-particle abrasion process in this study was carried out using Al_2O_3 particles (50 μm) that were directed perpendicularly at a distance of 10 mm for 10 seconds and at a compressed air pressure of 400 kPa. Suggestions for further investigations include changing the airborne-particle abrasion protocol by modifying particle size, abrasion time, distance, and pressure and by evaluating how that can affect the strength of FSZ differently. Nonetheless, airborne-particle abrasion affects the strength of FSZ and PSZ differently. Clinicians should be aware that, combined with zirconia's vulnerability to LTD, airborne-particle abrasion may promote premature failure in the large numbers of these restorations being placed.

Low-temperature degradation has been expressed as a concern with zirconia dental restorations based on the failures of the zirconia hip prostheses reported early in the 2000 decade.[16] However, the authors are unaware of any clinical evidence in dentistry. Nevertheless, this slow but autocatalytic phenomenon is well established. Zirconia grain size, sintering temperatures, cubic phase, and yttrium distribution, combined with a moist environment, all play a role in zirconia's relation to LTD.[3] In this study, both zirconia types were subjected to an artificial aging procedure in a steam autoclave at 125°C under 200 kPa pressure for 8 hours. Because the thermal activation energy required for the tetragonal to monoclinic phase transformation is ∼106 kJ/mol, it is estimated that 1 hour of steam autoclave treatment at 125°C under 200 kPa has the same effect as 1 year in vivo.[19] Therefore, the 8 hours of artificial aging performed in this study approximated 8 years of clinical service. Whether that is an accurate approximation may be debatable. Nevertheless, neither zirconia material displayed any sign of meaningful change in flexural strength after artificial aging, supporting LTD resistance in these materials. The results of this study are in agreement with those of other studies,[20,21] where autoclave aging procedures of zirconia material induced higher phase transformation from tetragonal to monoclinic but did not affect the flexural strength of the zirconia ceramic.

The zirconia material in this study was not subjected to mechanical or thermomechanical cycling, which may result in a different interpretation of the results. Further investigations into combining multiple treatments of FSZ versus PSZ, such as staining, airborne-particle abrasion and then thermomechanical cycling, are recommended to determine how that can affect both FSZ and PSZ. Finally, the conclusion, that vacuum-based sintering provides minimal benefits to the flexural strength of zirconia, was confirmed with the artificial aging procedures. All the specimens were sintered prior to aging in a vacuum-based furnace with no significant enhancement to the flexural strength compared with those sintered in a regular sintering furnace.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The flexural strength of FSZ was approximately half that reported for PSZ.
2. The sintering of zirconia restorations should follow the manufactures instructions, with no significant benefit for the use of vacuum-based furnaces over regular sintering furnaces.
3. Staining enhanced the flexural strength of FSZ, with no effect on the flexural strength of PSZ.
4. Airborne-particle abrasion can lower the flexural strength of FSZ, while enhancing the flexural strength of PSZ. However, further research is required before any clinical recommendations can be made.
5. Artificial aging has no effect on the flexural strength of either FSZ or PSZ.

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