

## **Shear Bond Strength of Different Zirconia Generations Bonded with Self Adhesive Resin Containing MDP**

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**Statement of Problem:** Do the different zirconia generations respond similarly to the same bonding protocol of self-adhesive resin containing MDP.

**Materials and Methods:** Four groups of zirconia specimens were used (n=10 each): according to the generation of zirconia used: group Hi Translucent is generation 1, group Super Translucent is generation 2, group Top Translucent is generation 3, and group Esthetic Explore is generation 5. Ten zirconia cuboids were dry-milled of each of the 4-zirconia blocks using a CAD CAM milling machine then sintered in a ceramic oven. Forty composite cuboids 4X4 were fabricated and cemented using self-adhesive resin cement containing MDP to each zirconia cuboid using custom constructed Plexi-Frames. All specimens were thermally cycled and subjected to shear bond strength in the universal testing machine.

**Results:** The mean SBS was higher in EX group (30.58+ 11.9) followed by HT group (23.16+ 3.6), then TT group (21.59+ 6.6), and finally ST group (21.47+ 6.1). One-Way ANOVA revealed a significant difference in SBS between tested groups.

**Conclusions:** The 5th generation of zirconia scored the highest mean SBS values. More surface treatment protocols should be suggested for each zirconia generation.

**Keywords:** Zirconia, generations, SBS, Shear bond strength, and surface treatment.

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## Introduction

The most often prescribed ceramics for posterior crowns are monolithic or full-contour zirconia prostheses. (1) This rapid switch to zirconia monolithic prosthesis was enhanced by the high clinical accomplishment and longevity of zirconia posterior ceramic crowns, precise milling, and increasing metal alloy prices. zirconia in the past: is known as strong but ugly, with limited indications, and impossible to bond. It continues to improve, from opaque and tough to better esthetics and more translucent but weaker ceramics. These advances in esthetics and translucency, increased flexural strength, results in a growing list of clinical applications, and a much better understanding of the chemistry and bonding options of zirconia. Dentists nowadays need to be aware of the qualities, traits, and variations across zirconia generations since these factors will determine whether a prosthesis is successful or unsuccessful. (2)

Yttria-stabilized zirconium oxide ceramics, also known as Yttria-stabilized Tetragonal Zirconia Polycrystal (Y-TZP or zirconia), could efficaciously replace the classic Porcelain-fused-to-metal (PFM) restorations. This growing success of Y-TZP is principally owed to the unique combination of high mechanical properties (3,4) and superior esthetics. Adding to this are the superior biological characters (3,4), proven clinical success rates (5,6), precise milling process (7,8), decreased wear to antagonists (9,10), as well as reasonable material and fabrication costs. (11,12)

The addition of Yttria helps stabilize the tetragonal phase of zirconia through a phenomenon known as “transformation toughening”.(13-16) The amount of Yttria content in zirconia would dictate the mechanical and optical properties. It should be noted that while the optical qualities are improved by adding more yttria, the strength and toughness are somewhat compromised.

Zirconia generations can be categorized based on how much Yttria they contain (mol percent). (2,17)

The first generation conventional 3Y-TZP zirconia contains 3 mol% Yttria. 3Y-TZP is partly alleviated in the tetragonal phase and has the highest fracture toughness and flexural strength, as well as the highest opacity, among other generations. (18,19) This renders it preferable mainly as frameworks in bi-layered long-span or posterior fixed dental prostheses (FDP) with higher occlusal loads.

By raising the Yttria concentration to 4 and 5 mol percent, new generations of zirconia have been created to improve aesthetics and are used for full-contour monolithic restorations. Yttria lowers the coefficient of heat expansion and enlarges the zirconia particle size. These generations are more transparent and less prone to low-temperature deterioration than the opaque traditional 3Y-TZP because they include between 30 and 50 percent cubic polycrystals. (20,21) However, because of the enhanced cubic phase, reduced porosity, and larger cubic grains, the light transmission rose by 43 to 45 percent. (11)

According to Rosentritt, et al, in 2019, (22) we could distinguish 5 generations of zirconia:

1st Generation 3Y-TZP (opaque, flexural strength > 1000), 2nd Generation 3Y-TZP (5 % more translucent, flexural strength 900 MPa), 3rd Generation 5Y-TZP (15 % more translucent, flexural strength 600 MPa), 4th Generation 4Y-TZP (10 % more translucent, flexural strength 750 MPa) and 5th Generation 3Y/4Y/5Y-TZP (1-15 % more translucent, flexural strength 550–1200 MPa) (Multilayer with translucency gradients).

Transformation toughening is a crucial component for better flexural strength, but it is not present in the compositions of these two generations, limiting their toughness and flexural strength. This constricts their clinical suggestion to a single unit, partial coverage,

and short-span FDPs in zones of restricted occlusal load. (23-25)

However, the stubborn nature of zirconia to bonding which is owed to its glass-free polycrystalline structure has always been its utmost clinical challenge. Many dental clinicians still doubt the bond ability of zirconia to resin composite cements up to the present moment, and some still question the role of zirconia primer in the bonding procedure. Nonetheless, there is quite evidence in the published literature proving the strength and reliability of zirconia bonding to resin composites after different surface treatment protocols including airborne-particle abrasion, tribochemical silica airborne-particle abrasion, low-fusion porcelain application, hot chemical etching solutions, selective infiltration etching, laser irradiation, plasma spraying, and zirconia ceramic powder coating. (2,13-16)

The introduction of 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) molecule has awakened the hopes for better bonding results. Since then, there has been a notable agreement in literature, including many systematic reviews, that the use of MDP-based primers on sandblasted zirconia surfaces significantly improves their bond strength to resin composites. (26-31) Hereby, the long-standing battle between the misconceptions of clinicians versus evidence-based researchers regarding the bond ability of zirconia sounds remains unresolved.

The questions remained around zirconia primers; about their effectiveness, reliability, and the amount of bond increase versus the cost and complexity of the conditioning procedure, especially

This research was conducted to assess if the SBS of the currently available types of zirconia with different translucencies will respond similarly to the zirconia primer.

The null hypothesis of this study was that, the SBS of the 4 tested zirconia generations would not be affected by using zirconia primer.

## Materials and Methods

### *Specimen grouping:*

From the data of a previous study, (31) a power analysis was done to determine the number of specimens that would be required in each test group to determine statistical differences between the groups. Based on this analysis 4 groups (n=10) were tested: (HT, ST, TT and EX). Four groups of zirconia specimens were created (n=10 each): according to the generation of zirconia used: group HT is generation 1 is (HT, Shenzhen Upcera Dental Technology CO., Ltd.), group ST is generation 2 (ST, Shenzhen Upcera Dental Technology CO., Ltd.), group TT is generation 3 (TT, Shenzhen Upcera Dental Technology CO., Ltd.), and group EX is generation 5 (Esthetic Explore, Shenzhen Upcera Dental Technology CO., Ltd.). (Table 1)

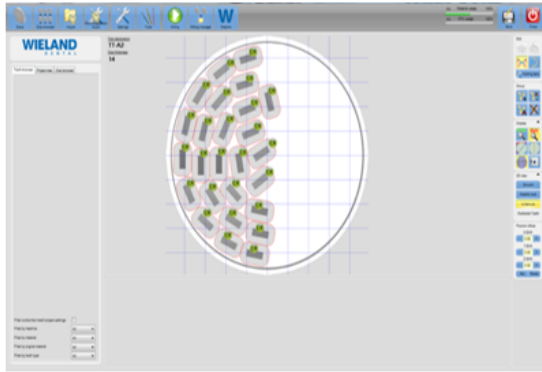
Table (1): The zirconia generations used in the study.

Groups	Translucency	Strength
HT 3Y-TZP	39%	1200 MPa
ST 3Y-TZP	43%	1200 MPa
TT 5Y-PSZ	47%	1000 MPa
EX 4Y-PSZ-5-YPSZ	1 <sup>st</sup> layer 49% 2 <sup>nd</sup> layer 48% 3 <sup>rd</sup> layer 47.5% 4 <sup>th</sup> layer 46.5% 5 <sup>th</sup> layer 46%	1 <sup>st</sup> layer ≥ 600 MPa 2 <sup>nd</sup> layer ≥ 675 MPa 3 <sup>rd</sup> layer ≥ 750 MPa 4 <sup>th</sup> layer ≥ 850 MPa 5 <sup>th</sup> layer ≥ 900 MPa

### *Zirconia samples fabrication:*

The cuboid samples were designed on AutoCad software (Autodesk, San Francisco, California), with the dimension of 6x6 mm with 3mm thickness, then exported as STL file (standard triangulation language) file to CAD software (Dwos software, Weiland Dental, Germany). For the EX (Multilayered group) the cuboids were cut perpendicular to the layers orientation. (Fig.1) 10 Cuboids were dry-milled of each of the 4-zirconia blocks using a CAD/CAM milling machine

(Weiland Zenostar coping, Weiland Dental, Germany). Forty cuboids were collected and each group of 10 was separated and marked to avoid faulty mixing between groups. (Fig.2)



**Figure 1:** Nesting of zirconia cuboids in the block.



**Figure 2:** Milled HT zirconia block.

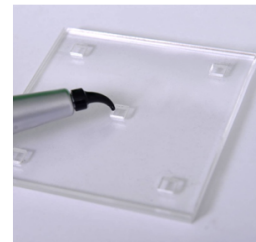
After milling, the samples were thoroughly cleaned with jets of air and then placed in the ultrasonic solution for 1 min and left to dry. The cuboids were then sintered in a ceramic oven (Multimat Easy, Degudent, Dentsply, Hanau, Germany). Each group exactly following their manufacturer's sintering protocol to preserve their physical, mechanical, and optical properties. Cuboids were left to cool down slowly and collected, each group in its dedicated dry clean container.

### **Resin samples fabrication:**

A 3 mm thickness transparent Plexi-frame was laser cut to form 4X4 cubic housings. (Fig.3) A second Plexi-frame was placed under it to act as a base. Composite resin (Sonicfill, KERR BENELUX, Rue d'Artagnan 28/2, 4600 Visé, Belgique) was injected in 4X4 housing. (Fig.4) A glass microscope slide was positioned on top of the composite to ensure a flat composite surface. Forty composite cuboids were then light-cured for 40 sec. on all surfaces. (32) After curing, the composite was pushed from the housing created. (Fig.5)



**Figure 3:** Laser cut 4X4 cubic housing in Plexi-frame.



**Figure 4:** Injecting composite to obtain composite resin blocks.



**Figure 5:** composite cuboids.

**Surface treatment of zirconia samples:**

Each zirconia cuboid was air particle abraded (APA) by 50 $\mu$  Al<sub>2</sub>O<sub>3</sub> particles (Zest Dental Solutions Global Corporate Headquarters, 2875 Loker Avenue East, Carlsbad, CA 92010, USA). A custom-made jig was formed to normalise a 60° angle and 1 cm distance between the blasting tip and the zirconia surface. (33) A pressure 0.2MPa was advised for better bonding. (34) Cuboids from each group were collected and placed in an ultrasonic solution for 1 min then removed and left to dry.

**Surface treatment of Composite samples:**

Using active brushing with a microbrush, Silane coupling agent (3M Silane, 3M, ESPE, USA) was applied to all composite cuboids bonding surfaces and thinned out by soft jets of air.

**Bonding Composite samples to Zirconia samples:**

A 3 mm thickness transparent Plexi-frame was laser cut to form 6X6 cubic housings. It acts as a housing for the zirconia cuboids during bonding. Zirconia cuboids were secured in the 6X6 Plexi housing and the composite cuboids were secured in the 4X4 Plexi housing. Resin cement (TOTALC-RAM, Itena Clinical, Central Parc Bat B - 97 Allée de la Louve, 93420 – Villepinte, France) was applied to the zirconia abraded surface, and the 4x4 Plexi housing containing composite cuboids were inverted on 6x6 Plexi housing. A weight of 5kg was placed on top of 4X4 Plexi housing for 5min the load was applied on a group of 4 samples. Light curing was applied using LED (1500 mW/cm<sup>2</sup>, Eighteen Curing Pen, Sifary medical technologies, Jiangsu Province, China) for 40 seconds. After setting the two Plexi-frames were separated and specimens were collected and stored in distilled water at 37°C for 24 h. (35) (Fig.6)

**Specimen testing:**

Specimens were secured longitudinally in the lower member of the

universal testing machine (Instron 8874, Instron Corp.). An unbeveled force application tip was applied to the zirconia-cement interface at a cross-head speed of 0.5mm/min until a sudden drop of the load was recorded in Newton. Shear bond strength was calculated from the equation: SBS (MPa) = load (N)/area (mm<sup>2</sup>). (37)

**Statistical analysis:**

Kolmogorov-Smirnov normality tests were considered to evaluate the normality of the data distributions. One-way ANOVA tests were used to analyze the statistical significance between tested groups. Values were presented as mean  $\pm$  standard deviation. All statistical analysis was conducted using SPSS v. 17 (BM Corp; Armonk, NY). P-value < 0.05 was considered statistically significant (S) and p-value  $\geq$  0.05 was considered statistically nonsignificant (NS). Charts were created using Microsoft Excel 2013. An alpha level of 0.05 was used as a decision point for statistical significance.

**Results****1) Kolmogorov-Smirnov normality tests**

The test was applied to 4 groups to determine the normality of the distribution of the results in each group. This selects the statistical analysis applied.

The results of the test showed no significant difference between data in each group. So, data are normally distributed in each group.

**2. Descriptive Statistics:**

Analysis showed significantly higher mean shear bond strength of EX group (30.58+ 11.9) than all the other groups. The difference between HT group (23.16+ 3.6), TT group (21.59+ 6.6), and ST group (21.47+ 6.1) was non-significant. Analysis of Variance (ANOVA) - One-Way ANOVA revealed a significant difference between tested groups (P= 0.0358) (Table 2)

**Table 2:** Descriptive Statistics with One-Way ANOVA results.

Variables	Mean	±SD	ANOVA	
			F	P value
Group Ex	30.58 <sup>A</sup>	11.9	3.1706	0.035
Group ST	21.47 <sup>B</sup>	6.1		
Group TT	21.59 <sup>B</sup>	6.6		
Group HT	23.16 <sup>B</sup>	3.6		

Different superscript capital letter in the same column indicating statistically significant difference (p < 0.05) \*; ns; non-significant (p > 0.05)

## Discussion

The current study investigated the shear bond strength of 4 different zirconia generations treated with MDP-containing primer and cemented with the same protocol.

Long time ago, zirconia has been considered as one type of ceramics and up-to-authors-knowledge, all previous studies addressed zirconia as such.

Till now zirconia has been developed into 5 generations (22) and they differ from each other in composition; the amount of alumina, Yttria, Cubic zirconia, and stabilizing materials; Yttria, Alumina, Calcia, Ceria or Magnesia that have been added to the material to partially stabilize tetragonal phase. Hafnia could be added as well to the material to increase the total volume of transformation toughening. On top of that, zirconia generations vary greatly in flexural strength, toughness, esthetic qualities, translucency, and hence indications. (2)

During planning for the methodology workflow in the current study, many measures were put in place to help standardize the study's aim while also safely protecting it. Instead of the more common circular discs, square-shaped cuboids were created to prevent application-tip slippage during SBS testing. For the multilayered EX blocks cutting the samples were made perpendicular to the layering direction to simulate the clinical application where all the layers are represented in the bonded surface. Plexi-frames were used and laser-cut to precisely aid in the fabrication of composite cuboids. In addition, they secure the cuboids during bonding and aid in the perfect

positioning of cuboids. On top of that, they secure specimens to the universal testing machine during shear force application.

The fitting surface of zirconia samples were air abraded prior to adhesive resin application. This approved to increase the retentive surface, also air abrasion claimed to cause phase transformation from the tetragonal to the monoclinic phase, which causes an increase in volume and hence suppresses the expansion of scratches. This transformation is theorized to enhance the damage tolerance of zirconia ceramic material. (33-37)

Results indicated a statistically significant difference in SBS between testing zirconia-tested generations. With the highest mean of SBS earned by the 5th generation. These findings may dictate further investigations aiming at finding better bonding protocols dedicated to each zirconia generation.

As confirmed by previous studies that the application of priming agents containing MDP enhanced the bond strengths of resin-based luting agents to high translucent zirconia material. The results support those of earlier research that employed zirconia ceramics that were more often used. Those studies found that using a hydrophobic phosphate monomer (MDP) improved the binding performance between zirconia ceramics and resin-based luting agents. (36-39)

Larsson, et al in 2019 (40) however, disproved the idea that transparent zirconia had a stronger bond strength than opaque zirconia since the outcomes from both materials were equal.

Whereas the more Yttria containing zirconia 4 Y-TZP is zirconia containing 4 mol % and 5 Y-TZP zirconia contains 5 mol % owing to the Ultra-high translucency and Supra-high translucency, showed lower SBS with the lowest score by the supra-high translucency.

The higher shear bond strength obtained with EX group may be related to the higher strength of the 4 mol % zirconia layer which was not weakened by sandblasting (41) while for the other groups, cohesive failure within the sandblasted surface may contributed to the weaker shear bond strength.

In addition, the variations in crystalline structure in the multi-layered Esthetic Explore may create more retentive surface compared with the other groups. They have demonstrated a considerable change in shear bond strength to composite after treating their sandblasted surfaces with the zirconia primer. And thus, the null hypothesis to this research was rejected.

### Conclusion:

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

- 1) Generations 5 EX (Esthetic Explore) produce stronger shear bond strength than the other generations.
- 2) Shear bond strength is significantly affected by zirconia generations.

### Recommendations

Further investigations are recommended either to support or reject current claims.

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