

Effect of Zirconia Type on Its Bond Strength with Different Veneer Ceramics

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Keywords

Zirconia framework; coloring pigments; microtensile bond strength; veneer ceramics; liner material; surface treatment.

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Abstract

Purpose: The bond strength between veneer ceramic and the zirconia framework is the weakest component in the layered structure. This bond was proven to be sensitive to the surface finish of the framework material and to the type of the veneer ceramic and its method of application. New colored zirconia frameworks were introduced to enhance the final esthetics of the layered all-ceramic restoration. The aim of this study was to investigate the effect of zirconia type, white or colored, and its surface finish on the bond strength to two veneer ceramics.

Materials and Methods: Five commercial zirconia framework materials (Cercon white and yellow, Lava white and yellow, Procera zirconia) received either of the following surface treatments: CAD/CAM milled surface, airborne-particle abrasion, and liner application. Two veneering ceramics were used to veneer the specimens: Noble Rondo and Ceram Express. The disc-shaped layered specimens were cut into microbars, and microtensile bond strength (MTBS) test was conducted. Structural and chemical differences between the white and colored frameworks were evaluated using scanning electron microscopy (SEM) and energy dispersive analysis. Two-way ANOVA and Tukey post hoc tests were used to analyze the data (p < 0.05 was considered significant).

Results: The type of zirconia framework had a significant effect on the core–veneer bond strength, which was material dependent. The bond strength to colored zirconia was significantly weaker compared to white zirconia frameworks. Different surface treatments had different effects on the core–veneer bond strength according to the zirconia material used. Although no marked chemical differences between the examined zirconia materials could be found, there were structural differences, especially between white and colored zirconia and for different zirconia frameworks of different manufacturers, which significantly affected core–veneer bond strength values.

Conclusion: The addition of coloring pigments to zirconia frameworks resulted in structural changes that require different surface treatment before veneering. To prevent delamination and chipping failures of zirconia veneered restorations, careful selection of both framework and veneer ceramic materials, in addition to proper surface treatment, are essential for maintaining good bond strength.

Core veneered restorations are the cornerstone for prosthetic dentistry, and the combination of a strong core and an esthetic veneer ceramic has proven successful for many decades. The need for superior esthetics and biocompatibility led to a material shift, as all-ceramic core materials are currently replacing dental casting alloys, but the principle itself remains the same.¹

Due to strength limitations, application of all-ceramic core materials was limited to three- or four-unit fixed partial denture restorations and where gnathologic conditions, like the occlusal relation and functional stresses, are optimal. The introduction of tetragonal zirconia polycrystals (TZP) as a restorative core material opened the design limits of all-ceramic restorations to extensive multiunit reconstructions with high confidence and success rates. The unique chemical stability, the superior mechanical properties, and the esthetic color, combined with CAD/CAM technology all make zirconia the core material of choice.²

Aboushelib et al

To gain the strength benefits of the core material, the coreveneer bond strength must be of adequate strength and toughness to transmit functional stresses from the esthetic veneer to the underlying framework. In a previous study, the zirconiaveneer bond strength was inferior compared to other all-ceramic systems, which suggests that the layered zirconia frameworks are more susceptible to delamination and chipping under function.³ In a following investigation, the bond strength of different layering and press-on veneer ceramics to one type of zirconia substrate was studied, and it was observed that the zirconiaveneer bond strength was material dependent. Additionally, it was reported that surface treatment, such as airborne-particle abrasion or the application of liner material, had significant effect on the bond strength and the mode of failure.⁴

Different zirconia framework materials are now available on the market and despite sharing a relatively similar chemical structure, their fabrication technique, milling procedure, and sintering temperature differ, and thus the manufacturers' recommended surface treatments differ significantly. It has been shown that the final product is influenced by all these variables.^{5,6}

Although zirconia frameworks are more esthetically acceptable compared to metallic frameworks, zirconia is in practice still too white and too opaque. Therefore, colored zirconia frameworks were introduced to improve the overall color matching result of the restoration.² Different techniques, such as adding metallic pigments to the initial zirconia powder before or after pressing the milling blocks, dipping the milled frameworks in the dissolved coloring agents, or application of liner material to the sintered white frameworks, were used for coloring zirconia frameworks.^{3,7} Direct advantages of colored zirconia frameworks are the reduction in veneer thickness required to mask the white color of the underlying framework⁸ and the discard of the masking liner material, which is applied before layering the veneer ceramic. The application of the liner material was proven to have a detrimental effect on zirconia veneer bond strength, especially when using press-on ceramic.⁴

The aim of this work was to investigate the effect of the structural differences between different zirconia framework materials and the effect of different surface treatments on their bond strength to two veneer ceramics. Additionally, the effect of coloring pigments on the microscopic structure of zirconia frameworks was also investigated.

Materials and methods

Twenty-five discs of different zirconia framework materials received either of the following surface treatments: airborneparticle abrasion, liner application, or unchanged CAD/CAM milled surface. The discs were veneered with two types of veneer ceramics and cut into microbars to measure the coreveneer microtensile bond strength (MTBS). Structural and chemical differences were analyzed with scanning electron microscopy (SEM) and element diffraction analysis (EDX).

Preparation of the specimens

Five commercially available zirconia framework materials were selected. All zirconia framework materials were milled to disc forms $(19.4 \times 3 \text{ mm}^2)$, and sintered in the equipment advised by

the manufacturers. The following surface treatments were applied: (i) milled surface unaltered, (ii) airborne-particle abraded with aluminum oxide particles ($120 \mu m$) at 3.5 bar pressure, or (iii) airborne-particle abraded and coated with the liner material supplied by the veneer manufacturers. One layering ceramic (Nobel Rondo, Nobel Biocare AB, Goteborg, Sweden) and one press-on veneer ceramic (Cercon[®] Ceram Express, Degudent GmbH, Hanau-Wolfgang, Germany) were selected based on their previously measured superior bond strength to a zirconia framework material.³ Material properties are summarized in Table 1.

In the groups where a liner material was applied, a glass instrument was used to apply a single thin continuous layer of the material, which was ultrasonically condensed, dried, and fired according to manufacturers' recommendations (Austromat 3001, Dekema Dental-Keramiköfen GmbH & Co, Freilassing, Germany) and finally airborneparticle abraded with 50 μ m glass particles at 1.5 bar. For the layering ceramic, the zirconia discs were seated in an adjustable aluminum mold, and the ceramic slurry was condensed, blot dried, pneumatically pressed, and fired according to the manufacturer-prescribed sintering cycle. For the press-on veneer ceramic; a wax disc $(19.4 \times 3 \text{ mm}^2)$ was fused to the surface of the zirconia, and the specimen was processed according to manufacturers' recommendations for pressing technique. Five specimens were prepared for each of the 30 test groups (5 zirconia frameworks/2 veneers/3 surface treatments).

Microtensile bond strength test (MTBS)

The disc-shaped layered specimens of each test group were cut into microbars ($6 \times 1 \times 1 \text{ mm}^3$) using a diamond-coated saw under water cooling (Ecomet, Buehler, Ltd., Evanston, IL). The microbars were examined using a stereomicroscope (SZ Olympus, Tokyo, Japan) at magnification of 25× to detect surface defects, and only sound microbars were selected. Randomly, 18 sound microbars were selected from each group. According to previously reported methods, the microbars were attached to the attachment unit using an adhesive resin (Clearfil SE Bond, Kuraray Medical, Inc., Okayama, Japan) and were loaded to failure at a crosshead speed of 1 mm/min (Instron 6022, Instron Limited, High Wycombe, UK). The maximum load at failure was extracted from computer-generated files.^{9,10}

Scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX)

The zirconia side of the tested microbar was ultrasonically cleaned and gold sputter-coated for SEM examination. Fracture was classified as cohesive in the veneer ceramic or interfacial across the core–veneer interface. Highly polished zirconia specimens were also prepared and examined to measure grain size, the location of coloring pigments, and to examine the surface morphology. Additionally, the density, the hardness, and the surface roughness after different surface treatments were examined using Archimedes method, Vickers hardness test, and surface profilometery, respectively. The Ra value, which is

	Material	Manufacturer	Thermal expansion coefficient (ppm/°C)	Weight content (oxygen was a balance)		
Zirconia framework materials	White Cercon base	Degudent GmbH,	10.5	Zirconium 52% to 59%; oxygen 26% to 34%; yttrium 5% to 7%; hafnium 3% to 5%;		
	Yellow Cercon base	Hanau-Wolfgang, Germany	10.5			
ma	White Lava	3M ESPE, AG, Germany	10.0	trace elements alumina, silica, and		
Zirconia framework	Yellow Lava		10.0	sodium 0.8% to 1.63%; coloring		
	Procera zirconia	Nobel Biocare AB, Goteborg, Sweden	10.4	pigments for shaded frameworks: iron 0.2% to 1.6%, magnesium 0.2% to 0.4 traces of erbium 66, cerium, and praseodymium		
amics	Nobel Rondo zirconia dentine	Nobel Biocare AB, Goteborg, Sweden	9.3	Fine-grained homogeneous feldspathic porcelain for layering technique		
aer cer	Nobel Rondo zirconia base liner	Nobel Biocare AB, Goteborg, Sweden	9.3	Fine-grained homogeneous feldspathic porcelain		
Vene	Cercon Ceram Express	Degudent GmbH,	10.5	Glass ceramic for pressing technique		
	Cercon Ceram Express liner	Hanau-Wolfgang, Germany	10.5	Feldspathic porcelain		

Table 1 Material properties

the arithmetic average of the recorded surface elevations and depressions, was used as a parameter to compare the surface roughness of the examined specimens (SJ-400, Mitutoyo Corporation, Kawasaki-shi, Japan).¹¹ The distribution of the stabilizing elements and the coloring pigments and the basic chemical structure of each of the used zirconium framework materials were investigated using EDX (EDAX, Inc., Mahwah, NJ), enabling analysis of parts of materials with a resolution of approximately $0.5 \,\mu$ m.

Statistics

The MTBS was statistically analyzed by two-way ANOVA followed by Tukey post hoc test with the zirconia type and the veneer/pretreatment as variables (SigmaStat Version 3.0,

SPSS, Inc, Chicago, IL). Considering the available resources, the sample size (n = 18) was selected based on a power analysis study (1 – β = 1) where the selected significance level (α = 0.05) and a large effect size difference (F = 0.4) would result in clinically relevant interpretations.

Results

The MTBS values and the failure pattern of the different zirconia and veneer/pretreatment groups are summarized in Tables 2 and 3 and graphically depicted in Figure 1. Statistical analysis revealed a significant difference between MTBS values between the different zirconia (F = 8.9, p < 0.001) and veneer/pretreatment groups (F = 12.4, p < 0.001) and their

Table 2 Microtensile bond strength (MPa) of the different zirconia types

	Nobel Rondo			Cercon Ceram		
	As-milled	Airborne-particle abraded	Liner	As-milled	Airborne-particle abraded	Liner
Cercon white	36.5 (9.5) ^{ABbc}	42.4 (11.5) ^{Bc}	28.5 (15.3) ^{Aab}	22.8 (2.0) ^{Aa}	37.9 (5.1) ^{Cbc}	31.6 (7.7) ^{Aabo}
Cercon yellow	31.6 (7.7)Abc	24.3 (8.7) ^{Aab}	29.3 (7.3)Abc	25.9 (12.7) ^{ABab}	17.2 (5.3) ^{Aa}	37.1 (12.2) Ad
Lava white	24.8 (6.3) ^{Aa}	29.7 (7.3) ^{Aab}	23.4 (11.4) ^{Aa}	23.0 (8.1) ^{Aa}	36.1 (8.1) ^{Cbc}	41.8 (9.4)Ac
Lava vellow	30.1 (6.9) ^{Abc}	20.8 (10.3) ^{Aab}	29.4 (7.4) ^{Abc}	26.4 (11.5) ^{ABab}	16.8 (5.2) ^{Aa}	39.3 (7.7) ^{Ac}
Procera*	30.8 (10.4) ^{Aa}	49.8 (25.8) ^{Bb}	31.9 (12.8) ^{Aa}	33.9 (5.6) ^{Bab}	39.1 (8.2) ^{Aba}	25.8 (8.5) ^{Ba}

No significant differences were observed for different zirconia frameworks if the mean MTBS has the same lowercase letter.

No significant differences were observed for one zirconia framework regarding veneer/pretreatment if the mean MTBS has the same uppercase letter.

*Commercial Procera frameworks are delivered already airborne-particle abraded, so additional surface treatment is not necessary.

	Nobel Rondo			Cercon Ceram		
	As-milled	Airborne-particle abraded	Liner	As-milled	Airborne-particle abraded	Liner
Cercon white	65% cohesive	100% cohesive	80% interfacial	60% cohesive	100% cohesive	65% interfacial
Cercon yellow	60% interfacial	85% interfacial	90% interfacial	55% cohesive	70% interfacial	70% interfacial
Lava white	90% interfacial	80% cohesive	70% interfacial	90% cohesive	70% cohesive	70% interfacial
Lava yellow	80% cohesive	100% interfacial	70% interfacial	70% cohesive	80% interfacial	75% interfacial
Procera	100% interfacial	80% cohesive	85% interfacial	80% cohesive	90% cohesive	90% interfacial

Table 3 Failure types and percentage of the different zirconia types

interaction (F = 5.4, p < 0.001). The results of the post hoc analysis are summarized in Table 2.

Regarding surface treatment, special attention should be paid to airborne-particle abrasion as a pretreatment. The bond strength of the airborne-particle abraded Cercon white, Lava white, and Procera with both veneers was generally stronger than the as-milled group of the same manufacturer. In contrast, the bond strength between airborne-particle abraded Cercon yellow and Lava yellow with both veneers was significantly lower than the as-milled group of the same manufacturer. Furthermore, comparing Cercon white and yellow and Lava white and yellow in the airborne-particle abrasion group, yellow frameworks had significantly lower MTBS values compared to white frameworks in the Cercon Ceram specimens.

The application of a liner material for both veneers was basically beneficial for the colored zirconia frameworks in addition to white Lava, with Cercon Ceram giving the highest



Figure 1 Microtensile bond strength (MPa) of the tested groups.

MTBS values. On the other hand, using a liner often increased the percentage of interfacial failure compared to the airborne-particle abrasion groups of the same test combination (Table 3).

SEM analysis revealed that different zirconia framework materials had different structural compositions at the microscopic grain level. Figure 2A demonstrates the homogenous grain structure of white Lava framework. Average grain size was estimated to be 0.5 μ m. Addition of coloring pigments to the same material resulted in changing the grain structure of the material to elongated tubular form (Fig 2B) with deposition of the coloring pigments at grain boundaries and surfaces (Fig 3A). Occasionally, thick deposits of these metallic coloring pigments were observed to completely mask the underlying Lava zirconia framework material (Fig 3B). Procera framework material demonstrated a larger and a unique biconcave granular structure, which is less densely packed (Fig 4A). The grain size, structural density, and the location of the coloring pigments are summarized in Table 4.

The effect of surface finish on the surface architecture can be demonstrated by comparing the as-milled surface with the abraded surface (Fig 4B,C) where the milling trace lines were eliminated after airborne-particle abrasion. Additionally, airborne-particle abrasion resulted in material loss, creation of sharp cracks, and indentations, which were more aggressive in colored framework materials compared to white ones (refer to surface roughness in Table 4). On the other hand, airborneparticle abrasion reduced the percentage of interfacial failure of white framework materials (Fig 4D).

EDX analysis demonstrated minor chemical differences between the used zirconia framework materials. The weight percentage of yttrium, as a stabilizing element, ranged between 5% and 7% for the tested frameworks, while the basic coloring pigment was ferric oxide in addition to other trace elements all contributing to less than 1% of the weight of the examined colored frameworks (Table 1). It was observed that the concentration of the coloring pigments was higher at the grain boundaries, which was at the expense of the concentration of the stabilizing elements.

Discussion

Core-veneer bond strength was previously estimated using different approaches and techniques that actually recorded the fracture strength of the tested specimens rather than the



Figure 2 (A) SEM image of a white Lava framework material demonstrating typical grain structure; (B) surface structure of colored Lava framework. Addition of the coloring pigments by dipping technique resulted in elongated grain structures and surface lifts.

actual measurement of the bond strength. Direct interpretation of the obtained data was thus not straightforward.¹²⁻¹⁶ Using the MTBS test has its advantages. The applied test force is perpendicular to the tested interface, and the small size of the tested microbars reduces the chance of incorporation of structural flaws, which resulted in a more accurate estimation of the core-veneer bond strength with less scattering of data.¹⁷ On the other hand, using MTBS test with dental ceramics requires careful handling of the specimens to avoid creation of structural defects.

As failure of a layered structure is expected to occur in the weakest material or in the weakest interface of the system, the inferior zirconia veneer bond strength was an observation of interest. Manufacturers' and researchers' efforts focused on increasing the strength of the core and the veneer ceramic materials, while the bond between them was not adequately considered.¹⁸

Previous investigators observed chipping and delamination failures for veneered zirconia restorations during different loading tests, which was a point of consideration indicating that more light should be shed on the relation between the core and the veneer.^{14,19,20} The core–veneer bond strength was previously investigated using MTBS, and the results indicated that the bond strength was sensitive to the surface finish of the framework material and to the type of the veneer ceramic and its method of application.

The results of this study demonstrated that the core–veneer bond strength is also affected by the type of zirconia framework material and its surface finish. While the CAD/CAM milling process produced trace lines on the surface, airborne-particle abrasion resulted in erasing these lines and in the creation of a rougher surface. For white framework materials, the obtained surface roughness (Ra = 4 to 6 μ m) reduced the percentage of interfacial failure (Fig 4D), and enhanced MTBS values. On the other hand, it resulted in the creation of sharp scratches and larger defects on the surface of yellow frameworks with higher surface roughness values (Ra = 5 to 9 μ m) (Fig 4C). This observation could explain the reduction in MTBS values and the high percentage of interfacial failure observed for the abraded yellow frameworks (Table 3).



Figure 3 (A) Deposition of coloring pigments at grain boundaries and on top of grain surfaces in colored Lava framework; (B) crystallization of dipping solution pigments on the surface of Lava framework observed occasionally on the surface of the tested specimens.



Figure 4 (A) Surface structure of Procera zirconia framework demonstrating wide inter-globular spaces as result of the used die pressing technique; (B) surface landmarks of CAD/CAM milling process of Procera zirconia framework; (C) SEM image showing scratches and pitting of yellow zirconia framework materials after airborne-particle abrasion,

Careful attention should be paid to the liner material. It was reported that applying this masking material over a smooth zirconia surface before pressing the veneer ceramic significantly weakened the core–veneer bond strength and increased the chances of interfacial failure.⁴ In this study, the liner was applied over an airborne-particle abraded surface, and even though it increased the bond strength with colored zirconia (D) interfacial fracture was observed to occur from the globular surface of nonabraded Procera framework material. Zirconia was directly observed at fracture surface.

frameworks, it also increased the percentage of interfacial failure. For white frameworks, its application resulted in reducing the core–veneer bond strength and increasing the percentage of interfacial failure (Tables 2 and 3).

Even though the EDX analysis of all tested framework materials revealed a typical chemical structure of TZP with a yttrium concentration between 5% and 7%,²¹ further analysis revealed

Zirconia framework	Average grain size (µm)	Density (g/cm ³)	Location of pigments	Milled surface landmarks	Surface roughness after airborne-particle abrasion (Ra)	Surface hardness (VHN)
White Cercon base	0.3	6.07	None	Parallel lines for CAM version and radial lines for CAD version	4 to 6 μ m	1330
Yellow Cercon base	0.6	6.07	Grain boundaries	1	6 to 9 µm	1240
White Lava	0.5	6.08	None	Smooth surface	4 to 6 µm	1335
Yellow Lava	0.58	6.07	At grain surfaces and boundaries		5 to 9 µm	1244
Procera	0.3 to 1.6	5.8	None	Concentric and radial lines	4 to 6 µm	1245

Table 4 Average grain size, density, pigment location, and surface roughness of the tested zirconia framework materials

that different zirconia framework materials had different surface and bulk structure characteristics. Differences in grain size, shape, composition, density, and hardness indicated that different surface treatment methods would result in differences in the final structure (Table 4). An observation of interest was that the increase of the concentration of the coloring pigments at grain boundaries was at the expense of the stabilizing element. Reduction in the percentage of the stabilizing elements would result in higher percentage of tetragonal-monoclinic transformation, which if occurring on the surface of the framework, would result in grain pullout and surface lifts, which may explain the surface characteristics of the colored frameworks (Figs 2A,B).^{2,22}

A possible explanation for this finding is that the melting point of ferric-oxide (1565°C), the main coloring pigment, is much lower than the melting point of yttrium and hafnium oxides (2410 and 2751°C, respectively), which would result in competitive displacement of the stabilizing elements by the metallic pigments in the liquid state, which can occur during sintering the zirconia frameworks.²³ The concentration of the detected trace elements erbium, cerium, and praseodymium could not be accurately calculated using EDX technology and requires other analytical methods. It was previously proven that slight alteration of the concentration or the location of the stabilizing elements has direct effect on the mechanical properties of the zirconia framework.^{21,22,24} A fatigue process was previously described to start on individual areas on the surface, leading to monoclinic spots, resulting in microcracking and surface lifts. This process then extends slowly deeper toward the bulk of the material, and the presence of coloring pigments at grain boundaries combined with reduction in yttrium concentration may significantly affect such process.25,26

A point worth noting is that even though that both colored Cercon and Lava specimens demonstrated chemical and structural similarities, the coloring method used during fabrication of the milling blocks differs for each system. For Cercon, the pigments are added before isostatically pressing the milling blocks, which guarantees homogenous and equal distribution of the pigments. On the other hand, the Lava system depends on dipping the milled framework in the coloring solution where the required pigments diffuse through the whole structure. A problem associated with such a method is that the concentration of the pigments is higher on the outer surface compared to the bulk of the framework. It was also observed that the surface pigments tend to crystallize on the surface of the framework during the sintering process (Fig 3B). This crystallized layer could be responsible for weakening the bond with the veneer ceramic.

Conclusion

Proper selection of veneer ceramic and zirconia framework material and proper surface treatment are of extreme importance to guarantee that zirconia veneered restorations will perform according to their expected functional demands.

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