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Research Article

Effect of Thermal Cycling on Shear Bond Strength between Zirconia Core and Ceramic Layering Under Different Surface Treatments - An Invitro study

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Abstract

Background: Durable bonding between a zirconia framework and veneering ceramic remains important because veneer chipping and interfacial delamination can compromise bilayered zirconia restorations. This in vitro study evaluated whether different zirconia surface treatments and thermal cycling influence core-veneer shear bond strength and failure pattern.

Materials and Methods: Forty computer-aided design/computer-aided manufacturing zirconia cylinders (5 mm diameter, 3.5 mm height) were randomly allocated to four surface conditions (n = 10): untreated control, 110 micrometre aluminium-oxide air abrasion followed by 5% hydrofluoric-acid conditioning, diode-laser irradiation, and erbium, chromium:yttrium-scandium-gallium-garnet laser irradiation. Standardized veneering-ceramic cylinders (2 mm diameter, 2 mm height) were fired onto the treated surfaces. Each treatment was divided into non-thermocycled and thermocycled subgroups (n = 5). Thermal aging was performed between 5 degrees Celsius and 55 degrees Celsius. Shear bond strength was measured at 1 mm/min, and failure modes were evaluated by scanning electron microscopy. Mann-Whitney U, Kruskal-Wallis, and chi-square tests were used at a significance level of 0.05.

Results: Air abrasion plus hydrofluoric-acid conditioning produced the highest mean shear bond strength before and after thermal cycling (26.35 (SD 9.24) and 33.51 (SD 12.93) MPa), whereas diode-laser treatment produced the lowest values (12.03 (SD 3.81) and 11.18 (SD 5.95) MPa). Differences among surface-treatment groups were significant in the non-thermocycled ($p = 0.001$) and thermocycled ($p = 0.023$) conditions, but no within-treatment pre/post thermal-cycling comparison was significant. Failure pattern changed from predominantly mixed failure before aging to entirely adhesive failure after aging ($p < 0.001$).

Conclusion: Under the tested conditions, surface-conditioning strategy influenced measured zirconia-veneer bond strength more strongly than thermal cycling alone. The marked post-aging shift toward adhesive failure nevertheless indicates deterioration of interfacial integrity that was not captured by mean bond-strength values alone.

Keywords: Zirconia; Veneering Ceramic; Shear Bond Strength; Thermal Cycling; Surface Treatment; Laser Irradiation

Abbreviations

Al2O3: Aluminium Oxide; CAD-CAM: Computer-Aided Design and Computer-Aided Manufacturing; Er,Cr:YSGG: Erbium, Chromium-Doped Yttrium Scandium Gallium Garnet; HF: Hydrofluoric Acid; MPa: Megapascal; SBS: Shear Bond Strength; SEM: Scanning Electron Microscopy.

Introduction

Zirconia has become an established framework material for fixed prosthodontic restorations because of its strength, fracture resistance, biocompatibility, and compatibility with computer-aided manufacturing. In bilayered restorations, however, the clinical performance of the restoration depends not only on the zirconia substructure but also on the stability of the zirconia-veneering ceramic interface. Chipping and delamination can occur even when the zirconia framework itself remains intact [1-4].

Bonding at the core-veneer interface is multifactorial. Surface topography, wettability, interfacial contact, thermal-expansion compatibility, firing and cooling conditions, and the intrinsic properties of the veneering ceramic can all influence measured bond strength [1,2,5]. Airborne-particle abrasion is commonly used to create micromechanical irregularities, whereas laser irradiation has been investigated as a more controlled method of surface modification. Published results remain inconsistent, particularly because laser effects vary with wavelength, power, pulse characteristics, irradiation distance, and exposure protocol [6-8].

Thermal cycling is used in laboratory studies to challenge bonded interfaces by repeated exposure to temperature changes and moisture. The mismatch in thermal expansion and contraction between zirconia and veneering ceramic may generate interfacial stresses, and water penetration may further influence interface durability. Prior investigations have reported both significant and negligible effects of thermal aging, suggesting that aging response depends on material pairing and experimental protocol [3,9-11].

This study therefore compared four zirconia surface conditions - no treatment, aluminium-oxide air abrasion followed by hydrofluoric-acid conditioning, diode-laser irradiation, and erbium, chromium:yttrium-scandium-gallium-garnet laser irradiation - before and after thermal cycling. The primary outcome was shear bond strength; failure pattern after debonding was a secondary outcome. The null hypotheses were that surface treatment would not affect shear bond strength and that thermal cycling would not alter shear bond strength within each surface-treatment condition.

Materials and Methods

Study design and ethical clearance

This laboratory-based in vitro study used 40 zirconia specimens. Institutional ethical clearance was obtained from the Sri Ramakrishna Hospital Ethical Committee (EC/2024/0110/CR-64; approval date: 1 October 2024). The approved sample size was 40 specimens.

Zirconia specimen preparation and surface treatment

Forty CAD-CAM zirconium-dioxide cylinders, each 5 mm in diameter and 3.5 mm in height, were fabricated and sintered according to the zirconia manufacturer's instructions. The specimens were steam-cleaned and air-dried before treatment. They were randomly allocated to four surface-treatment conditions (n = 10 per condition).

Surface condition	Protocol
Untreated control	No surface treatment.
Air abrasion + HF	Air abrasion with 110 micrometre Al2O3 (Cobra, Renfert, Germany) for 1 minute, followed by 5% hydrofluoric acid (IPS Ceramic Etching Gel/Refill, Ivoclar Vivadent, Schaan, Liechtenstein) for 1 minute.
Diode laser	Surface irradiation with a PIOON H1 dual-wavelength diode laser under the standardized parameters used in the study.
Er,Cr:YSGG laser	Surface irradiation with a Waterlase iPlus Er,Cr:YSGG laser (BIOLASE, Irvine, California, USA), wavelength 2780 nm, under the standardized parameters used in the study.

Table 1: Zirconia surface-treatment protocols.

After surface treatment, all specimens were ultrasonically cleaned in distilled water and dried.

Veneering procedure

An aluminium mould with an internal diameter of 2 mm and a height of 2 mm was used to standardize the veneering ceramic. IPS e.max Ceram (Ivoclar Vivadent, Schaan, Liechtenstein) powder and liquid were manipulated at the manufacturer-recommended ratio and applied to the zirconia surface through the mould. Specimens were fired in a Programat P310 furnace (Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer-recommended firing schedule and cooled to room temperature.

Thermal cycling

Each surface-treatment condition was divided into a non-thermocycled subgroup and a thermocycled subgroup (n = 5 each). Thermal cycling was performed between 5 degrees Celsius and 55 degrees Celsius for 2000 cycles with a 15-second dwell time in each bath. After thermal cycling, specimens were stored in distilled water at room temperature until mechanical testing.

Shear bond strength and failure-mode assessment

Shear loading was applied at the zirconia-ceramic interface using an Instron ElectroPuls E3000 universal testing machine at a crosshead speed of 1 mm/min until debonding. Maximum load at failure was recorded in newtons and converted to megapascals by dividing the failure load by the bonded surface area. Debonded surfaces were examined by scanning electron microscopy (Zeiss Sigma VP FE-SEM) and classified as adhesive, cohesive within ceramic, cohesive within zirconia, or mixed failure.

Statistical analysis

Mean and standard deviation were calculated for each subgroup. Non-thermocycled and thermocycled values within each surface treatment were compared using the Mann-Whitney U test. The four surface treatments were compared separately under non-thermocycled and thermocycled conditions using the Kruskal-Wallis test. Failure-mode distributions were compared using the chi-square test. Statistical significance was set at $p < 0.05$.

Results and Discussion

Results

The air-abrasion-plus-HF condition had the highest mean SBS under both aging conditions, whereas the diode-laser condition had the lowest mean SBS. None of the four treatment-specific comparisons between non-thermocycled and thermocycled specimens was statistically significant. In contrast, the global comparison among surface-treatment conditions was significant both before thermal cycling ($p = 0.001$) and after thermal cycling ($p = 0.023$) (Table 2 and Figure 1).

Surface condition	Non-thermocycled, n	Mean +/- SD (MPa)	Thermo-cycled, n	Mean +/- SD (MPa)	Within-treatment p
Untreated control	5	25.19 +/- 7.45	5	24.24 +/- 10.76	0.841
Air abrasion + HF	5	26.35 +/- 9.24	5	33.51 +/- 12.93	0.421
Diode laser	5	12.03 +/- 3.81	5	11.18 +/- 5.95	1.000
Er,Cr:YSGG laser	5	22.84 +/- 18.68	5	21.18 +/- 8.66	0.691
Kruskal-Wallis p		0.001		0.023	

Table 2: Shear bond strength by surface treatment and thermal-aging condition. Within-treatment p values are from Mann-Whitney U tests; between-treatment p values are from Kruskal-Wallis tests.

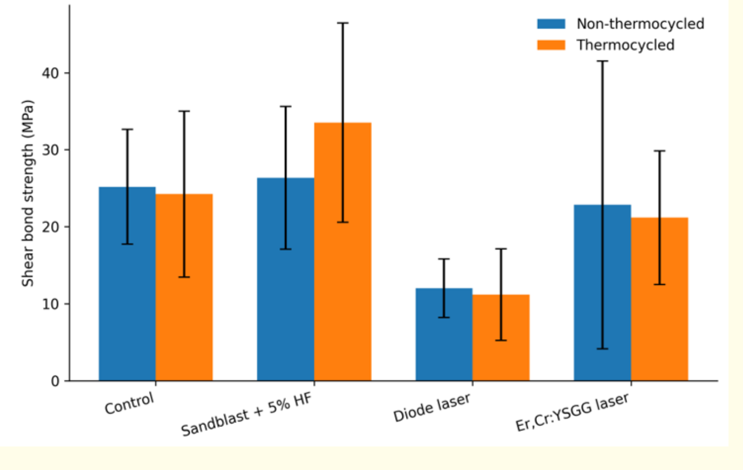

Figure 1: Mean shear bond strength (MPa) with standard-deviation error bars for each surface-treatment condition before and after thermal cycling.

Failure mode changed markedly after thermal cycling. Across the non-thermocycled specimens, 80% of failures were mixed and 20% were adhesive, with no cohesive failures. After thermal cycling, every specimen showed adhesive failure. The overall change in failure pattern was highly significant (chi-square = 26.67, df = 1, $p < 0.001$) (Table 3 and Figure 2).

Condition	Adhesive	Mixed	Cohesive
Non-thermocycled	20%	80%	0%
Thermocycled	100%	0%	0%

Table 3: Failure-mode distribution before and after thermal cycling.

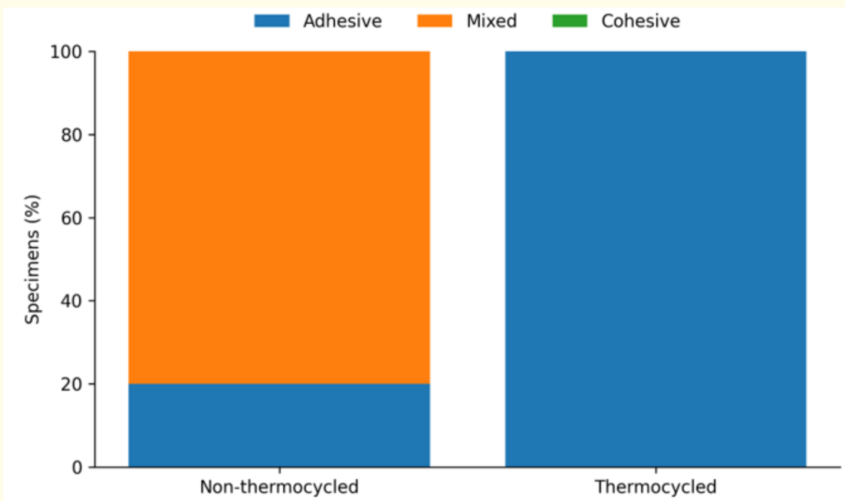

Figure 2: Distribution of adhesive, mixed, and cohesive failure before and after thermal cycling.

Discussion

The first null hypothesis was rejected because surface treatment significantly influenced SBS under both aging conditions. The air-abrasion-plus-HF protocol produced the highest mean values. Airborne-particle abrasion can increase surface area and create a more retentive topography, thereby improving micromechanical interlocking with the fired veneering ceramic. Similar advantages of abrasive conditioning have been reported in several zirconia-veneer studies, although the magnitude of benefit varies with the zirconia and veneering systems used [1,5,6,12,13].

The hydrofluoric-acid step requires cautious interpretation. Zirconia is a dense polycrystalline ceramic without the silica-rich glassy phase that makes conventional HF etching effective for glass ceramics. Consequently, the favorable performance of the combined protocol should not be attributed to classic HF etching of zirconia alone. In the present experiment, the acid step functioned as an adjunct after substantial mechanical roughening by 110 micrometre aluminium oxide. The result is therefore best interpreted as the performance of the combined protocol rather than evidence that HF alone is an effective zirconia treatment [12,13].

The diode-laser condition yielded the lowest SBS in both aging states. Laser conditioning is highly protocol-sensitive: wavelength, power, pulse mode, focal distance, irradiation time, and energy distribution determine whether useful microroughness or potentially unfavorable thermal alteration is produced. A systematic review found that laser irradiation can improve zirconia surface characteristics and bonding in selected protocols but also emphasized the absence of a single standardized laser protocol [8]. This helps explain why laser treatment should not be judged as a uniform category and why the diode protocol used here may have underperformed relative to airborne abrasion.

The Er,Cr:YSGG-treated specimens showed intermediate mean SBS values and substantial variability, particularly before thermal cycling. Experimental work using erbium-family lasers has similarly shown that treatment efficacy depends on energy settings and whether laser irradiation is used alone or in combination with another surface-conditioning method [7,14,15]. The present findings therefore do not establish that erbium-laser treatment is ineffective; rather, they indicate that the tested protocol did not outperform the conventional abrasive-conditioning strategy.

The second null hypothesis, concerning within-treatment SBS change after thermal cycling, was not rejected. Thermal cycling produced no statistically significant change in mean SBS within any treatment. Previous studies have reported mixed findings, ranging from clear aging-related reductions to minimal effects depending on the material combination and aging protocol [3,9-11]. The small subgroup size in the present study (n = 5) also limits power to detect modest aging effects and contributes to uncertainty around the numerical changes.

The failure-mode data add an important qualification to the SBS results. Despite the absence of significant within-treatment changes in mean SBS, thermal cycling shifted the fracture pattern from predominantly mixed failure to 100% adhesive failure. This suggests that thermal aging altered interfacial quality before a consistent reduction in measured failure stress became statistically detectable. Because SBS is a single end-point measure and can be influenced by specimen geometry and stress concentration, combining quantitative bond-strength testing with fracture analysis gives a more complete view of interface behavior [2-4].

The main limitations are the small thermocycled and non-thermocycled subgroups, the use of a single veneering ceramic, and the in vitro design, which did not reproduce simultaneous occlusal fatigue, pH changes, salivary proteins, or complex clinical loading. In addition, incomplete documentation of the exact laser operating parameters and air-abrasion pressure and distance restricts reproducibility of the conditioning protocols. Future studies should use larger groups, fully specified conditioning parameters, longer thermal aging, combined thermomechanical loading, and complementary surface characterization.

Conclusion

Within the limitations of this in vitro study, zirconia surface treatment significantly influenced core-veneer shear bond strength. Air abrasion with 110 micrometre aluminium oxide followed by 5% hydrofluoric-acid conditioning produced the highest mean bond strength under both non-thermocycled and thermocycled conditions, whereas diode-laser treatment produced the lowest values. Thermal cycling did not significantly alter mean shear bond strength within any individual surface-treatment condition, but it caused a highly significant shift to adhesive failure. Surface treatment therefore had the stronger measurable effect on bond strength, while thermal aging appeared to compromise interfacial integrity in a way that was more clearly detected by failure-mode analysis than by mean shear bond strength alone.

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