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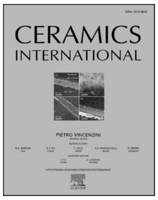

Short-pulse direct laser writing of 3Y-TZP zirconia: A superior surface modification strategy for enhanced resin bonding and adhesion durability

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ABSTRACT

The clinical success of zirconia-based restorations relies on surface modifications that enhance adhesion without compromising material integrity. This study evaluates Direct Laser Writing (DLW) as a non-contact, contamination-free alternative to conventional grit-blasting methods (alumina particle abrasion [SB] and silica-coated alumina treatment [SC]. Surface roughness (R_a), wettability, surface free energy (SFE), phase transformation, and shear bond strength (SBS) were assessed. Zirconia specimens ($N = 40$) underwent DLW at two distinct parameters (DLW35, DLW10) or conventional grit-blasting (SB, SC). Surface characterization included roughness measurements, contact angle analysis, and SFE calculation (Owens-Wendt method). Phase transformation was quantified using X-ray diffraction (XRD). SBS tests between zirconia and resin-matrix cement were performed under water storage and thermocycled conditions. Results showed DLW provided controlled, uniform roughness comparable to grit-blasting without inducing mechanical damage or contamination. DLW-treated surfaces exhibited significantly better wettability and higher SFE. SBS values were statistically similar across all groups, indicating stable adhesion performance under both dry and thermocycled conditions. DLW preserved the tetragonal zirconia phase substantially better (DLW35: 93 %, DLW10: 85 %) compared to grit-blasted surfaces (SB: 76 %, SC: 68 %), minimizing residual stress and microstructural damage. The study concludes DLW is a superior zirconia surface treatment, offering precise control, contamination-free surfaces, enhanced wettability, and maintained microstructural integrity, making it ideal for dental, maxillofacial, and orthopedic applications.

1. Introduction

The demand for dental restorations is increasing due to rising aesthetic expectations and growing awareness of dental health [1]. Patients seek materials that not only restore function but also blend seamlessly with natural dentition [2]. In recent years, the field of dentistry has been striving to become metal-free to address concerns related to metal allergies, hypersensitivity, and the aesthetic limitations of metal-based restorations [3]. Zirconia has emerged as a highly favorable material in restorative dentistry, offering significant advantages over traditional titanium implants and restorations [4,5]. Its superior aesthetic qualities, biocompatibility, and resistance to corrosion make zirconia an ideal choice for dental applications [6,7] Unlike titanium, zirconia closely mimics the natural tooth color, providing enhanced aesthetic outcomes [8]. Zirconia is widely used in crowns, bridges, inlays, onlays, veneers, implant abutments, and orthodontic brackets, benefiting from its mechanical properties and aesthetic appeal [9].

Moreover, the strength and chemical stability of zirconia are crucial for its performance in the oral environment, where it must endure strong occlusal forces and a complex chemical environment [4]. Zirconia's high fracture toughness and resistance to wear ensure its durability under significant masticatory loads, while its chemical inertness protects it from degradation in the presence of oral fluids and varying pH levels [1,

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7]. Additionally, zirconia has become increasingly popular among patients who are allergic or hypersensitive to metals, as it provides a metal-free alternative that eliminates the risk of allergic reactions associated with metal restorations. This biocompatibility further drives the preference for zirconia in restorative dentistry [3,6]. However, the adhesion of resin cement to zirconia poses notable challenges due to its inherently inert and non-reactive surface [10,11]. Effective surface modification techniques are essential to improve this adhesion. Various methods such as air-borne particle abrasion, silica coating, electric discharge machining, plasma spraying, nano-silica coating, fluorination, hot acid etching and more recently laser-based techniques have been employed to enhance the surface characteristics of zirconia [12–20]. Traditional methods like air-borne particle abrasion and silica coating physically roughen the surface to improve mechanical interlocking but can introduce contaminants and result in inconsistent surface textures [21,22]. Additionally, air-abrasion can adversely affect the strength and microstructure of zirconia by inducing surface flaws and increasing susceptibility to low-temperature degradation, which can compromise the material's long-term performance [23,24]. Electric discharge machining (EDM) and plasma spraying alter the surface properties at a molecular level, offering improved adhesion but often involve complex and expensive procedures [16,25]. Nano-silica coating aims to enhance chemical bonding but may suffer from uneven coverage and durability issues [26]. Fluorination and hot acid etching modify the surface chemistry to increase reactivity but can be hazardous and may not produce adequate surface roughness for optimal bonding [14,27].

Among these methods, Direct Laser Writing (DLW) is a precise surface modification technique that utilizes focused laser beams to inscribe intricate patterns directly onto a material's surface, offering high precision and flexibility for creating detailed micro- and nano-scale structures without masks or templates [28,29]. DLW provides significant advantages over sandblasting for zirconia modification, including greater precision and control, minimized surface damage, enhanced customization, a cleaner process, and improved adhesion properties [30,31]. Different types of lasers, including CO_2 (pulsed and continuous wave), long- and short-pulse lasers like, Er,Cr:YSGG, Er: YAG, Nd:YAG and ultrashort pulse (femtosecond and picosecond) lasers, have been explored for their efficacy in modifying zirconia surfaces [32–39]. Ultrashort pulsed lasers, such as femtosecond lasers, offer significant advantages over long-pulse lasers in terms of the quality of the surface and topography. They create highly precise and controlled surface textures with minimal thermal damage, leading to superior microstructural integrity and enhanced adhesion properties[38,40–42]. In contrast, short-pulsed lasers are generally more cost-effective but can cause greater thermal damage, resulting in less optimal surface quality and potential structural compromises[38,41,43–45]. Despite the higher initial investment, the benefits of ultrashort pulse lasers in achieving superior bond strength and surface integrity make them promising options for advancing zirconia-based dental restorations [44,46–48]. The increasing use of zirconia in crowns, bridges, and implants reflects its growing importance in meeting the dual demands of durability and aesthetic excellence in modern dentistry [1,2].

For this study Shear bond strength (SBS) testing was conducted to assess the adhesive performance of the zirconia-resin cement bond under clinically relevant loading conditions [49]. Given that shear forces predominate in the oral environment, SBS serves as a reliable method for evaluating bond durability and the effectiveness of various surface treatments [50]. This study provides valuable insights into optimizing zirconia bonding strategies, ultimately contributing to improved adhesion longevity and the long-term success of dental restorations [51,52].

2. Materials and methods

In this study, 3 mol% yttria-stabilized zirconia (TZ-3YB-E, Tosoh Corporation, Tokyo, Japan) was used to fabricate zirconia substrates. This high-purity zirconia powder (99 % purity, 6.05 g/cm^3 density) consists of spherical granules (~60 μm in size), composed of ultrafine crystallites (~40 nm in diameter), ensuring a uniform and high-quality microstructure.

2.1. Zirconia (3Y-TZP) green compact disk preparation

Forty zirconia green compacts ($N = 40$) were fabricated using cold pressing techniques, with each experimental group consisting of 10 samples. Zirconia powder was carefully poured into a stainless-steel die (10 mm diameter, 30 mm height) and compressed under 200 MPa of pressure for 30 s, forming compact disks with final dimensions of 10 mm in diameter and 6 mm in height (Fig. 1).

2.2. Surface treatment of compact zirconia disks

The green compact disks were divided into four groups ($N = 10$ per group) based on the surface treatment applied. Two groups (DLW10, DLW35) underwent direct laser writing (DLW) for surface texturing, performed in the green state before sintering under the same conditions as the other groups. For the air abrasion groups, zirconia compacts were fully sintered in a high-temperature furnace (Zirkonofen 700, Zirkonzahn, Italy) at 1500 °C for 2 h in air, with a heating and cooling rate of 8.3 °C/min. After sintering, two groups were subjected to grit-blasting using alumina (SB) or silica-coated alumina (SC) air abrasion. Finally, all samples were ultrasonically cleaned in isopropyl alcohol for 2 min to remove surface residues (Fig. 1).

2.2.1. Alumina particle abrasion group (SB)

Fully sintered zirconia (Y-TZP) disks were subjected to controlled alumina (Al_2O_3) air abrasion using a COMPO-JECT™ gun dispenser (RØNVIG Dental Mfg. A/S, Denmark). The procedure was performed with 50 μm alumina particles at 2.5 bar pressure, maintaining a 10 mm distance between the disk surface and the nozzle tip. Abrasive treatment was applied for 20 s in a circular motion, with the nozzle positioned at a 45° angle to ensure uniform surface modification (Fig. 1).

2.2.2. Silica-coated alumina abrasion group (SC)

Similarly, fully sintered Y-TZP disks were treated with controlled abrasion of 30 μm silica-coated alumina particles (3M ESPE AG, Cojet sand, Germany) using a gun dispenser (COMPO-JECT™, RØNVIG Dental Mfg. A/S, Denmark). The abrasion was carried out at a constant pressure of 2.5 bar for 20 s, keeping the disk at 10 mm from the nozzle. The air abrasion was done in a circular motion, during which the angle of the nozzle was at 45° with respect to the Y-TZP disks (Fig. 1)

2.2.3. Short pulse direct laser writing groups (DLW10, DLW35)

Laser surface texturing for the DLW-treated groups was performed on green zirconia compact disks using an Nd:YAG laser (OEM Plus, SISMA, Italy), operating at a fundamental wavelength of 1064 nm, with an output power of 6 W, a spot size of 3 μm, and a pulse duration of approximately 35 ns.

Two distinct direct laser writing (DLW) patterns were applied.

- DLW10: The crossed-line patterns were spaced at 10 μm, with DLW referring to direct laser writing and "10″ indicating the pattern spacing (Table 1 and Fig. 1).
- DLW35: Similarly, DLW35 features a crossed-line pattern spaced at 35 μm (Table 1 and Fig. 1).

The laser texturing strategy and corresponding parameters are detailed in Table 1 and Fig. 1.

2.3. Surface characterization

The treated zirconia (3Y-TZP) surfaces were characterized as follows.

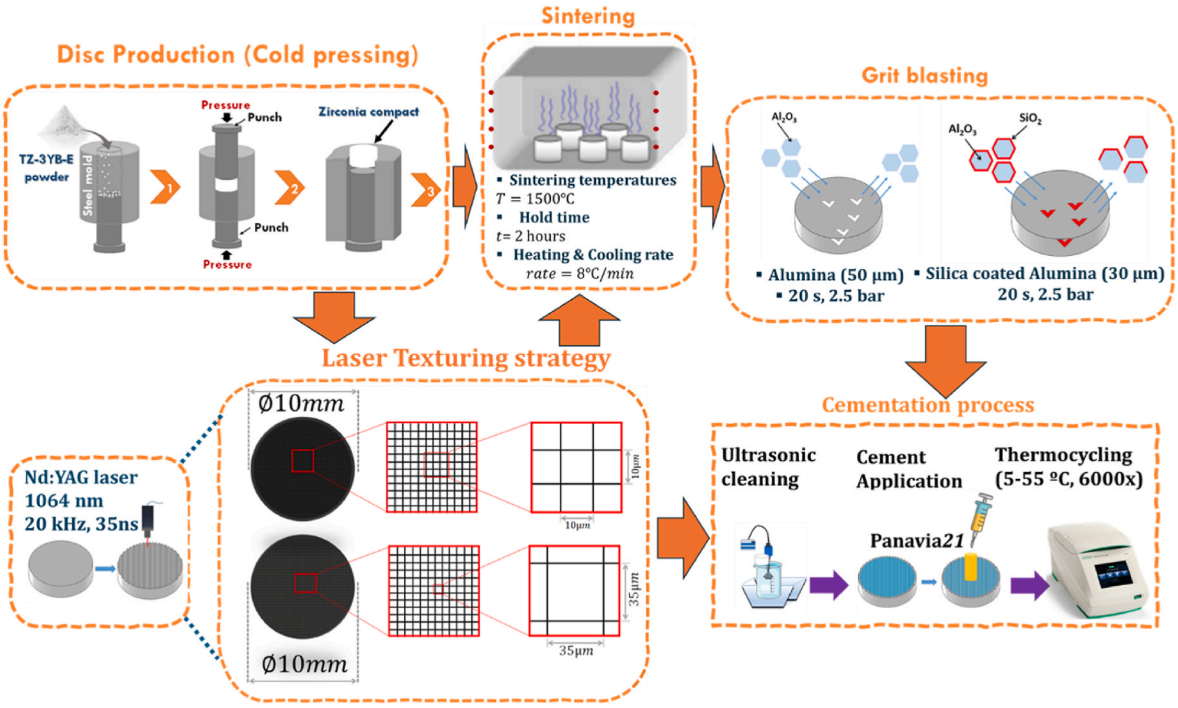

Fig. 1. – Schematic illustration of experimental workflow.

Table 1
Direct laser writing parameters for surface texturing of zirconia (green state).

Laser groups	Output power [W %]	Scanning velocity [mm/s]	Linear distance between crossed lines [μm]	Number of laser passes
DLW10	1	256	10	1
DLW35	10	128	35	1

2.3.1. *Surface roughness analysis*

Following surface modification, surface roughness measurements were performed for all groups using a contact profilometer (SurfTest SJ 201, Mitutoyo, Tokyo, Japan) equipped with a sharp diamond stylus (2 μm tip diameter). Ten measurements were taken at randomly selected areas on each sample, with a scanning speed of 0.5 mm/s and a sampling length of 0.8 mm. For laser-treated groups (DLW10 and DLW35), profilometry was conducted perpendicular to the texture direction to ensure accurate surface characterization. The measured roughness parameters included R_a (Arithmetic Average Roughness), a general measure of surface texture; R_q (Root Mean Square Roughness), which is more sensitive to larger deviations; and R_z (Average Maximum Height of the Profile), which captures extreme vertical differences on the surface.

2.3.2. *Micrographs of surfaces and interfaces*

All modified surfaces, zirconia-cement interfaces (cross-sectional samples), and fracture surfaces after adhesion testing were first sputter-coated with AuPd thin films and examined using an ultra-high-resolution Field Emission Gun - Scanning Electron Microscope (FEG-SEM) (NOVA 200 Nano SEM, FEI, Netherlands) for qualitative analysis. For zirconia-cement interface evaluation, zirconia bonded to resin-matrix cement samples were embedded in resin within a cylindrical tube and subsequently wet-ground on SiC papers (180–2000 grit) until a clean and smooth zirconia-cement interface was exposed.

2.3.3. *X-ray diffraction analysis (XRD)*

X-ray diffraction (XRD) analysis was performed to evaluate the crystalline phase composition and quantify monoclinic phase transformation after surface treatments. For each group (DLW35, DLW10, SB, SC), four independent zirconia specimens (N = 4 per group; total N = 16) were analyzed. Diffraction patterns were collected using a Bruker AXS D8 Discover diffractometer (Bruker, Germany) with Cu-Kα radiation (λ = 1.54060 Å). Data were recorded over a 2θ range of 27°–32°, at a step size of 0.04°, and a counting time of 1 s per step. Each specimen was mounted flat to ensure analysis of the treated surface, and the irradiated area was approximately 10 mm × 10 mm to provide representative sampling.

$$X_m = \frac{I_m(\bar{1}11) + I_m(111)}{I_m(\bar{1}11) + I_m(111) + I_t(101)} \tag{1}$$

$$V_m = \frac{1.311\ X_m}{1 + 0.311\ X_m} \tag{2}$$

Monoclinic and tetragonal phases were identified using X'Pert HighScore Plus software (PANalytical, Netherlands). The diffraction peak intensities for the monoclinic phase were represented as $I_m(\bar{1}11)$ at 28.2° and $I_m(111)$ at 31.5°, corresponding to the $(\bar{1}11)$ and (111) planes, respectively. The tetragonal phase was identified by the $I_t(101)$ peak at 30.2°, corresponding to the (101) plane. The volumetric fraction of the monoclinic phase (V_m) was calculated using the Toraya equation (Equation (2)), based on the integrated intensities of the relevant monoclinic and tetragonal peaks. All measurements were performed in triplicate for each specimen, and average values were used for quantitative analysis [6,53].

2.3.4. *Surface wettability*

The degree of wetting for all treated samples was evaluated through contact angle measurements using two liquid probes: deionized water (H_2O) and diiodomethane (CH_2I_2). The selection of these probes was based on the requirement of polar (H_2O) and nonpolar (CH_2I_2) contact angles for surface energy calculations.

Measurements were conducted at room temperature using the sessile drop method with an optical goniometer (OCA 15 Plus, Dataphysics, Germany). Droplets of ultrapure deionized water (18.2 MΩ cm) with a volume of 5 μL were dispensed at a dosing rate of 2.5 μL/s from a micrometric syringe, brought into contact with the surface, and allowed to stabilize for 15 s before measurement. Five readings were taken on each sample, and the average value was recorded as the final measurement. Prior to testing, all samples were ultrasonically cleaned in isopropyl alcohol for 1 min to remove surface contaminants [54].

2.3.5. *Surface free energy (SFE)*

The Owens-Wendt method was employed to estimate the surface energy components by analyzing the contact angles (θ) of two liquid probes—water (a polar liquid) and diiodomethane (a nonpolar liquid) on the solid surface. The calculations were performed using the following equations:

$$Cos(\theta_W) = 1 + \frac{2\gamma^d}{\gamma_w} - \frac{\gamma^p}{\gamma_w} \tag{1}$$

$$Cos(\theta_d) = 1 + \frac{2\gamma^d}{\gamma_d} - \frac{\gamma^p}{\gamma_d} \tag{2}$$

Where θ_W and θ_d represent the contact angles of water and diiodomethane on the solid surface, respectively, while γ_W and γ_d denote their corresponding surface tensions. The parameters γ^p and γ^d refer to the polar and dispersive components of water and diiodomethane on the solid surface.

By solving Equations (1) and (2) using contact angle data, γ^p and γ^d can be determined. The total surface energy (γ_s) is then calculated as $\gamma_s = \gamma^p + \gamma^d$. This method enables the quantification of surface energy components based on the wetting behaviour of polar and nonpolar liquids on the solid surface [55,56].

2.4. *Shear bond strength test*

2.4.1. *Cementation procedure*

Before cementation, the conditioned substrate was treated with ED Primer (Kuraray Noritake Dental Inc., Japan) according to the manufacturer's instructions. The primer was applied using a clean microbrush, allowed to react for the recommended time, and gently dried with air. A translucent polyethylene mold (inner diameter: 3 mm; height: 5 mm) was securely positioned on the primed substrate using a holder to ensure stability during cement application. A new polyethylene mold was used for each specimen. Dual-cure Panavia (Kuraray Co. Ltd, Tokyo, Japan) resin cement (see Table 2) was mixed according to the manufacturer's instructions and injected into the mold using a syringe (Centrix, DF, Rio de Janeiro, Brazil). Due to the precise fit of the mold and firm holder positioning, excess resin was minimal. Any remaining excess was carefully removed from the mold edges using a microbrush.

The cement was then photo-polymerized for 40 s from the top and four sides of each specimen. To prevent the oxygen-inhibition layer, an oxygen-inhibiting gel (Oxyguard II, Kuraray Co. Ltd, Tokyo, Japan) was applied to the exposed surfaces for 1 min before being thoroughly washed off. All surface conditioning and cementation procedures were carried out by the same calibrated operator to ensure consistency.

Following cementation, half of the specimens underwent dynamic aging (thermocycling: 5000 cycles, 5–55 °C, dwell time: 30 s) (Haake DC 10, Sigma-Aldrich, St. Louis, Missouri, USA) and were designated as the thermocycled group (TC). The remaining specimens were stored in distilled water at 37 °C for 24 h and classified as the water-stored group (WS).

Table 2
Chemical composition of Panavia F 2.0 (Kuraray Noritake Dental Inc. Japan) resin cement and primers.

Material	Chemical Composition
Resin-Matrix Cement: Panavia F 2.0 (Dual-cure Panavia, Kuraray Co. Ltd, Tokyo, Japan)	Methacryloxyethyl Trimellitate Anhydride (4-META), 10-Methacryloyloxydecyl Dihydrogen Phosphate (MDP), Bisphenol A Glycidyl Methacrylate (Bis-GMA), Tri-n-Butylborane (TBB), Catalysts, Silica Filler
ED primer	10-Methacryloyloxydecyl Dihydrogen Phosphate (MDP), HEMA (Hydroxyethyl Methacrylate), Water, Ethanol, Catalysts
Oxyguard II	Polyethylene Glycol (PEG)

2.4.2. *Shear bond strength testing*

To assess the bond strength between modified zirconia surfaces and resin-matrix cement, shear bond strength (SBS) tests were conducted. Specimens were securely mounted in the specimen holder of a Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany), and a force was applied to the adhesive interface until failure occurred. A 50 kgf load cell delivered the force as close to the substrate-adherend interface as possible at a crosshead speed of 1 mm/min. The resulting stress-strain curve was analyzed using TestXpert® software (Zwick ROELL, Ulm, Germany)

2.5. *Data analysis*

Statistical analysis was performed to assess differences in surface roughness, wettability, surface energy, and shear bond strength (SBS) among groups. Data normality and homogeneity were evaluated using the *Shapiro-Wilk* and Levene's tests. One-way ANOVA was applied for roughness, wettability, and surface energy when assumptions were met, while the *Kruskal-Wallis* test was used otherwise, followed by Tukey's HSD or *Mann-Whitney U* tests with Bonferroni correction for *post-hoc* analysis. Two-way ANOVA assessed the influence of surface treatment and aging (water storage vs. thermocycling) on SBS, with Tukey's HSD or for pairwise comparisons. Correlations between roughness, wettability, surface energy, and SBS were analyzed using Pearson's or Spearman's correlation coefficients. Statistical significance was set at $p < 0.05$.

3. Results

3.1. *Surface characterization*

A significant difference in surface roughness (R_a – average roughness, R_q – root mean square roughness, and R_z – average maximum height) was observed among the treated groups (one-way ANOVA, $p < 0.001$), as detailed in Table 3. *Post hoc Tukey's* HSD tests confirmed that DLW35 exhibited the highest roughness (R_a: 1.81 ± 0.23 μm, $p < 0.001$), followed by DLW10 (R_a: 1.02 ± 0.17 μm), both of which were significantly rougher than SB (R_a: 0.42 ± 0.02 μm) and SC (R_a: 0.34 ± 0.03 μm) ($p < 0.001$). No significant difference was found between SB and SC ($p = 0.687$), indicating that both grit-blasted surfaces exhibited comparable roughness as shown in Fig. 2. Although R_q and R_z values followed a similar trend, statistical analysis was conducted exclusively for R_a. These results demonstrate that Direct Laser Writing (DLW) treatments, particularly DLW35, produced significantly greater surface texturing than conventional grit-blasting, highlighting their potential for enhanced mechanical interlocking and adhesive bonding.

A significant difference in wettability, assessed through water and diiodomethane contact angles, was observed (Fig. 3) among the surface-treated groups (one-way ANOVA, $p < 0.001$ for both liquids), as presented in Table 4. *Post hoc Tukey's HSD* tests revealed that DLW10 exhibited the lowest contact angles for both water (53.40 ± 4.45°) and

Table 3
Surface roughness of all groups.

Groups	R_a (μm)	R_q (μm)	R_z (μm)
SB	0.43 ± 0.02	0.54 ± 0.03	3.13 ± 0.24
SC	0.46 ± 0.04	0.57 ± 0.05	3.03 ± 0.86
DLW10	1.02 ± 0.16	1.25 ± 0.23	6.05 ± 1.21
DLW35	1.81 ± 0.22	2.17 ± 0.30	10.26 ± 1.45

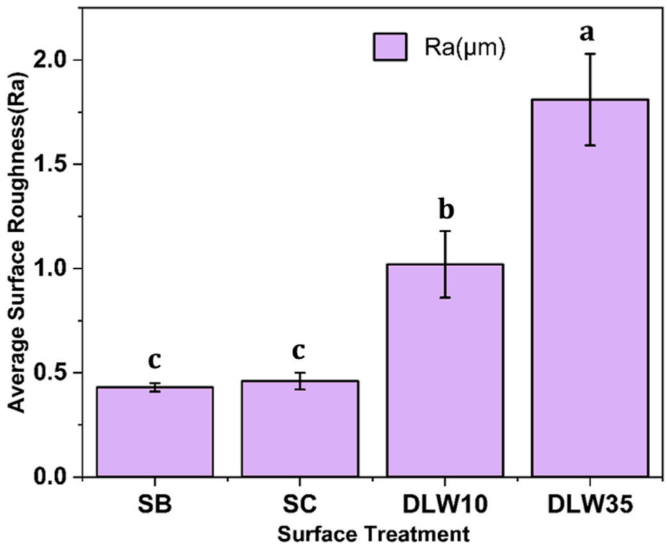

Fig. 2. Surface roughness (R_a) values across treatment groups. Groups with different letters indicate statistically significant differences ($p < 0.05$; *Kruskal-Wallis, Bonferroni-corrected*). SB and SC were not significantly different ($p > 0.05$).

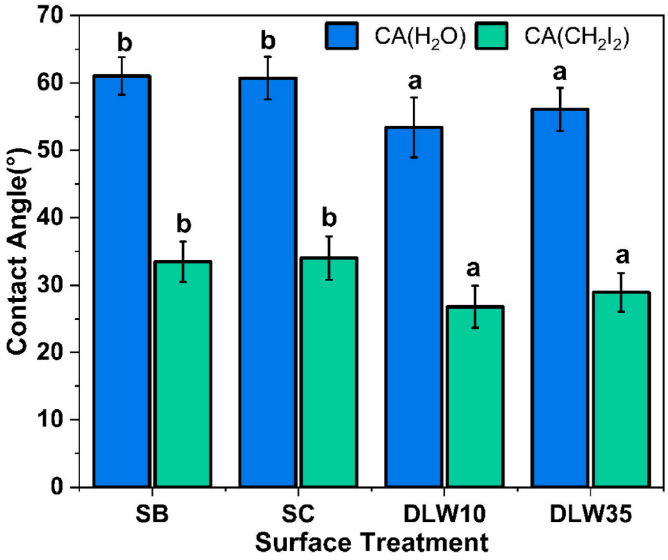

Fig. 3. Water and diiodomethane contact angles (mean ± SD) of zirconia after different surface treatments. Different letters above the bars indicate statistically significant differences among groups ($p < 0.05$, one-way ANOVA with Tukey's HSD).

Table 4
Water and diiodomethane contact angles and surface free energy (SFE, mean ± SD) of zirconia following different surface treatments.

Group	Water Contact Angle (°)	Diiodomethane Contact Angle (°)	SFE (mJ/m^2)
SB	61.03 ± 2.79	33.45 ± 3.01	53.57 ± 1.69
SC	60.72 ± 3.15	34.02 ± 3.22	53.56 ± 1.89
DLW10	53.40 ± 4.45	26.78 ± 3.12	59.38 ± 2.48
DLW35	56.07 ± 3.21	28.92 ± 2.87	57.43 ± 1.85

diiodomethane (26.78 ± 3.12°), indicating the highest wettability, followed by DLW35 (Water: 56.07 ± 3.21°, Diiodomethane: 28.92 ± 2.87°). Both laser-treated groups showed significantly greater wettability than SB (Water: 61.03 ± 2.79°, Diiodomethane: 33.45 ± 3.01°) and SC (Water: 60.72 ± 3.15°, Diiodomethane: 34.02 ± 3.22°) ($p < 0.001$), while no significant difference was found between SB and SC (water: $p = 0.843$, diiodomethane: $p = 0.764$), indicating similar wettability for both grit-blasted surfaces (Fig. 3). These findings highlight the superior wettability of DLW-treated surfaces, particularly DLW10, which achieved the highest hydrophilicity for both polar and nonpolar liquids. The lower contact angles observed in DLW-treated groups suggest a notable increase in surface energy, reinforcing their potential for applications requiring enhanced adhesion.

Surface free energy (SFE) values differed significantly among the surface treatment groups (one-way ANOVA, $p < 0.05$). The DLW10 group exhibited the highest SFE (59.38 ± 2.48 mJ/m^2), followed by DLW35 (57.43 ± 1.85 mJ/m^2), while the SB and SC groups showed markedly lower SFE values (53.57 ± 1.69 mJ/m^2 and 53.56 ± 1.89 mJ/m^2, respectively). Post-hoc Tukey's HSD analysis confirmed that both DLW10 and DLW35 had significantly higher SFE than SB and SC, whereas no significant difference was observed between SB and SC. The increased SFE in DLW-treated groups reflects enhanced surface wettability and suggests a more favorable surface environment for resin infiltration and chemical interaction, which are critical for durable adhesion. In contrast, the lower SFE values for grit-blasted groups (SB and SC) indicate relatively less wettable surfaces, potentially limiting the effectiveness of resin bonding. These results (Fig. 4) highlight the advantage of direct laser writing in producing zirconia surfaces with higher energy states, which may contribute to improved adhesive performance compared to conventional grit-blasting methods.

The measured surface free energy (SFE) values (Table 4) reflect meaningful differences in the surface chemistry and wettability produced by each treatment. Generally, a higher SFE indicates improved wettability, which can enhance the spreading and infiltration of resin cements and potentially facilitate stronger adhesive interactions at the interface. In this study, the DLW-treated groups showed SFE values comparable to or slightly higher than the grit blasting groups (SB & SC). This demonstrates the potential of DLW to create surfaces suitable for adhesive bonding. Nevertheless, the similarity in SFE values among all groups, along with comparable bond strengths, suggests that factors beyond SFE—such as chemical compatibility and micromechanical retention—also influence the final adhesive performance. These results highlight that while SFE is a useful indicator of surface wettability, it should be considered alongside other surface and interface

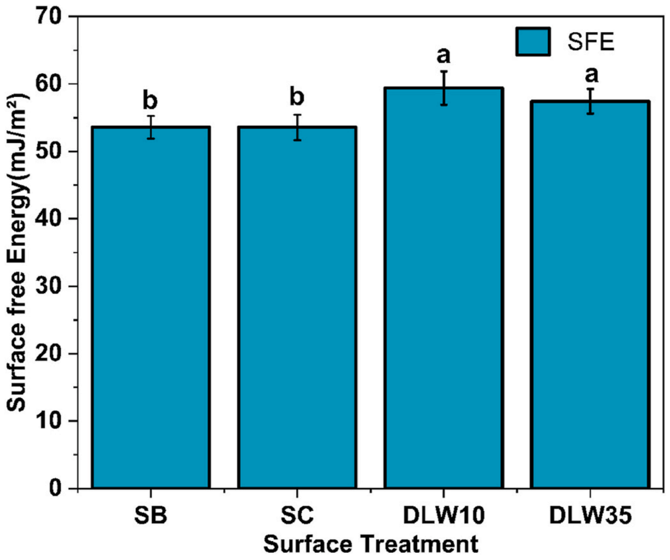

Fig. 4. Surface free energy (SFE, mean ± SD) of zirconia following different surface treatments. Different letters above the bars indicate statistically significant differences among groups ($p < 0.05$, one-way ANOVA with Tukey's HSD).

characteristics for a comprehensive understanding of adhesion mechanisms.

Phase composition (Fig. 5) analysis revealed distinct differences in tetragonal-to-monoclinic transformation among the various surface treatment groups. The DLW-treated groups demonstrated superior retention of the tetragonal phase, with DLW35 maintaining the highest tetragonal content at 93 % and only 7 % monoclinic transformation, while DLW10 retained 85 % tetragonal and 15 % monoclinic phase. In contrast, conventional grit-blasting methods induced more substantial phase transformation: the SB group showed 76 % tetragonal and 24 % monoclinic content, whereas the SC group exhibited the greatest transformation with 68 % tetragonal and 32 % monoclinic phase. These results indicate that direct laser writing, especially at higher processing parameters (DLW35), is considerably more effective at preserving the desirable tetragonal phase of zirconia compared to grit-blasting methods, which promote greater monoclinic phase formation and could potentially compromise the material's structural stability.

The SEM micrographs in Fig. 6 revealed distinct surface modifications induced by each treatment method. The SB and SC specimens exhibit irregular, randomly distributed roughness, characteristic of airborne particle abrasion. SB surfaces display deeper valleys and sharper depressions, resulting from the impact of larger alumina particles used in the grit-blasting process. In contrast, SC surfaces appear smoother and less textured, as the smaller silica-coated alumina particles (Rocatec™ system) create a more refined abrasion effect, modifying the surface through a combination of mechanical roughening and tribochemical interaction.

On the other hand, laser-treated surfaces (DLW10 and DLW35) demonstrated a well-controlled and uniform roughness, characterized by precisely structured periodic patterns created through Direct Laser Writing (DLW). The DLW35 group exhibits a distinct pyramidal surface architecture, evenly distributed across the material, while the DLW10 group presents a shallower but continuous surface texture with slightly less uniformity. These variations result from differences in laser processing parameters, particularly line spacing and scanning strategy, which influence material ablation and pattern formation.

These findings underscore a key difference between grit-blasted and laser-modified surfaces. While SB and SC treatments produce irregular roughness patterns, with SB showing greater surface disruption due to the larger alumina particles, DLW generates a precisely controlled microtopography without introducing foreign particles. The structured roughness achieved through DLW enhances mechanical interlocking and wettability, reinforcing its potential as a highly effective alternative for zirconia surface modification in adhesive applications.

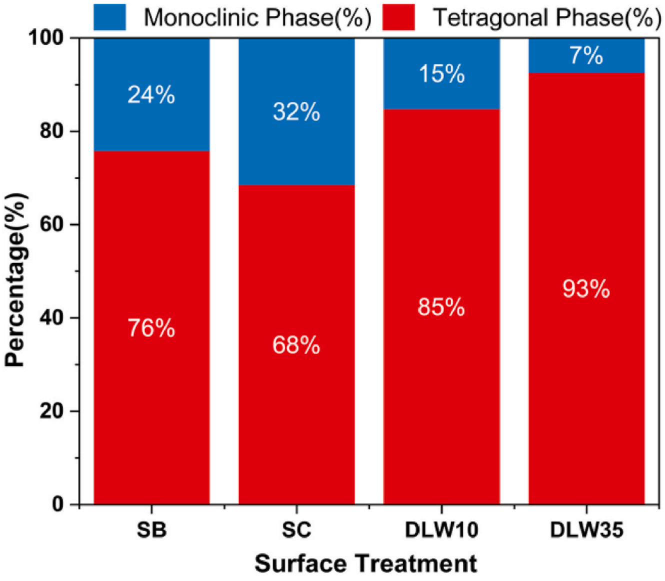

Fig. 5. Phase composition of zirconia (% tetragonal [t] and monoclinic [m]) after surface treatments (DLW35, DLW10, SB, SC). Results presented as stacked bar charts showing phase distribution for each group.

Fig. 7 presents the cross-sectional view of the zirconia-cement interfaces for samples subjected to different surface modifications. The resin-matrix cement is identifiable in micrographs as the darker phase layer, clearly delineating the interface. Importantly, there were no significant flaws detected at these interfaces, with a particular emphasis on the SB and SC groups. However, in the laser-textured groups, no defects were observed in the DLW10 specimens, and, as seen from the cross-sectional micrographs, the resin cement was able to flow into the shallow roughness. Unlike the rest of the groups, however, a clear narrow gap between the resin cement and zirconia surface can be observed in the cross-sectional micrographs of DLW35 specimen. The gap could possibly be present due to voids created due to air entrapment during cementation process. Although this single specimen does not showcase the conditions for all other specimens of DLW35 group, it cannot be ruled out either. This void could also be a factor for which, despite having higher surface roughness, DLW35 group could not produce higher bond strength compared to the rest of the groups.

3.2. Bond strength

3.2.1. Shear bond strength (SBS)

The sheard bond strength values obtained for the different surfaces are presented in Table 5 and Fig. 8. A two-way ANOVA revealed no significant differences in shear bond strength (SBS) values (Table 5) among the surface-treated groups in both dry and thermocycled conditions ($p > 0.05$). Post-hoc Tukey's HSD tests confirmed that in the dry condition, SB exhibited the highest SBS (9.91 ± 3.66 MPa), followed by DLW10 (9.15 ± 1.67 MPa), DLW35 (8.88 ± 2.35 MPa), and SC (8.49 ± 2.16 MPa), but the differences were not statistically significant ($p > 0.05$). After thermocycling, SBS slightly decreased across all groups, yet no significant difference was found between the initial and aged conditions ($p = 0.157$), indicating that thermocycling did not significantly degrade bond strength ($p = 0.249$). These findings suggest that laser-treated surfaces achieve bonding performance comparable to grit-blasted surfaces, reinforcing their suitability for long-term adhesive applications.

3.3. Mode of failure analysis

The SEM images of fractured surfaces for all groups, presented in Fig. 9, provide valuable insights into the failure mechanisms after shear bond testing. As observed, no significant remnants of resin-matrix cement were found on the zirconia surfaces across all groups, except for a single specimen (c6) from the DLW10 group, which retained a minor amount of cement, and small, scattered cement residues in a few SB specimens (a1, a2, and a3).

The absence of substantial resin remnants across the fractured surfaces indicates that the failure mode was predominantly adhesive in nature, occurring at the zirconia-cement interface rather than within the resin itself. This suggests that, regardless of the surface treatment method, the bonding mechanisms were primarily reliant on micromechanical interlocking rather than chemical adhesion. The slight presence of cement remnants in DLW10 and SB specimens may suggest areas of enhanced adhesion due to improved surface wettability or mechanical interlocking, yet this was not a widespread occurrence.

These findings reinforce the critical role of surface treatment in determining adhesive performance, as none of the tested groups exhibited cohesive failure within the resin cement, indicating that zirconia's inherent low surface energy remains a key challenge in achieving strong chemical bonding with resin-based adhesives.

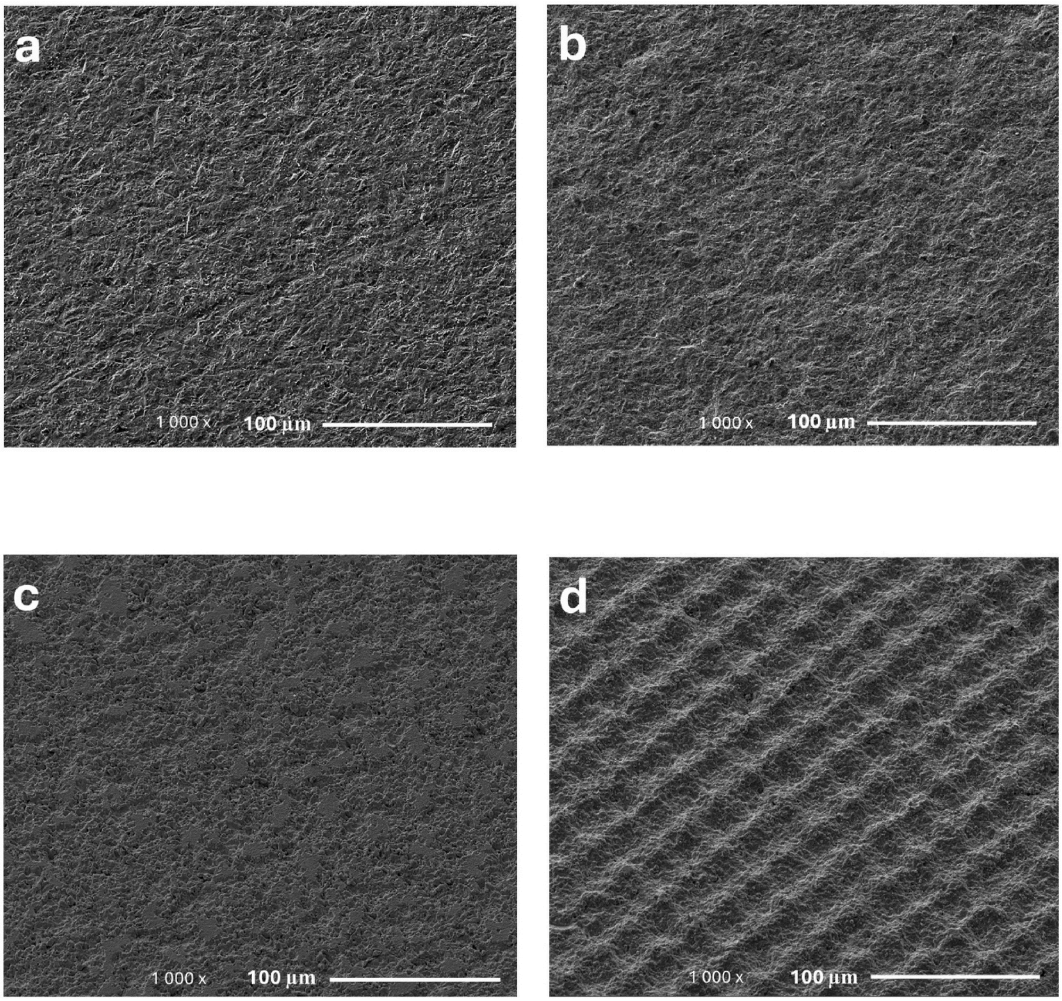

Fig. 6. SEM images (1000 × magnification) illustrating surface modifications on 3Y-TZP: (a) SB, (b) SC, (c) DLW10, and (d) DLW35.

4. Discussion

Zirconia (Y-TZP) excels in strength, toughness, fatigue resistance, and longevity, making it perfect for dental use [1,4]. Despite its mechanical prowess, it has generally weaker bond strength to resin-matrix cements compared to other ceramics [49,57]. Successful Y-TZP restorations rely on strong adhesion between zirconia, resin cement, and the tooth [58]. Thus, choosing the appropriate cement and luting method is essential [59]. This study's materials were selected for their representativeness and thorough validation, ensuring the findings are applicable to a wide range of similar materials [60].

A universally effective surface conditioning technique for all types of ceramics has not yet been developed [58,59]. The challenge lies in the numerous factors that influence the bond strength between resin-luting cements and ceramic materials [52]. Different ceramics possess unique properties, such as composition, surface texture, and chemical reactivity, which can significantly affect how well they bond with resin cements [10,61]. Consequently, dentists must have a thorough understanding of these ceramic characteristics and carefully select the most appropriate surface conditioning methods to optimize bonding outcomes [62,63]. This tailored approach is crucial for achieving durable and reliable restorations in dental practice [9,52].

This study explored Direct Laser Writing (DLW) using a short-pulse (ns-Nd:YAG) laser as a potential alternative to alumina blasting for zirconia surface treatment [19,64,65]. Various laser parameters were evaluated based on their influence on surface roughness, wettability (contact angles), and morphology, aiming to achieve comparable or improved performance relative to airborne particle abrasion while minimizing alterations to the material's microstructure [38,66]. Based on these evaluations, two laser-patterned surfaces, DLW10 and DLW35, were selected for further analysis. For comparison, conventional grit-blasting techniques were used as reference groups, including alumina particle abrasion (SB) and tribochemical silica-coated alumina treatment (SC, Rocatec™ system) [67,68].

To the best of the authors' knowledge, no previous study has systematically investigated the correlation between key surface properties of zirconia (3Y-TZP) post-surface modification, including surface roughness, wettability, and surface free energy (SFE), and their collective impact on bond strength with resin-matrix cement. This study aimed to bridge this gap by examining potential interdependencies among these surface characteristics and assessing their influence on the resulting adhesive performance [59].

Lasers such as CO_2, Er:YAG, Er,Cr:YSGG, and Nd:YAG have traditionally been employed for treating zirconia ceramics to boost bond strength [36,69–72]. The majority of the studies have shown positive outcomes in terms of bond strength between zirconia and resin cements, nevertheless conflicting results have also been reported[73–76]. Also, there is increasing interest in high-power lasers with ultra-short pulse durations, like, pico-, and femtosecond lasers, to further enhance the bonding of zirconia with composite cements [39–41,77]. Several studies have used Nd:YAG lasers, similar to the one used in this study, with different pulse duration/lengths [36,72,78–81]. Pulse durations holds importance since short-pulsed lasers bring distinct benefits, such as increased peak power and a smaller heat-affected zone, which allow for

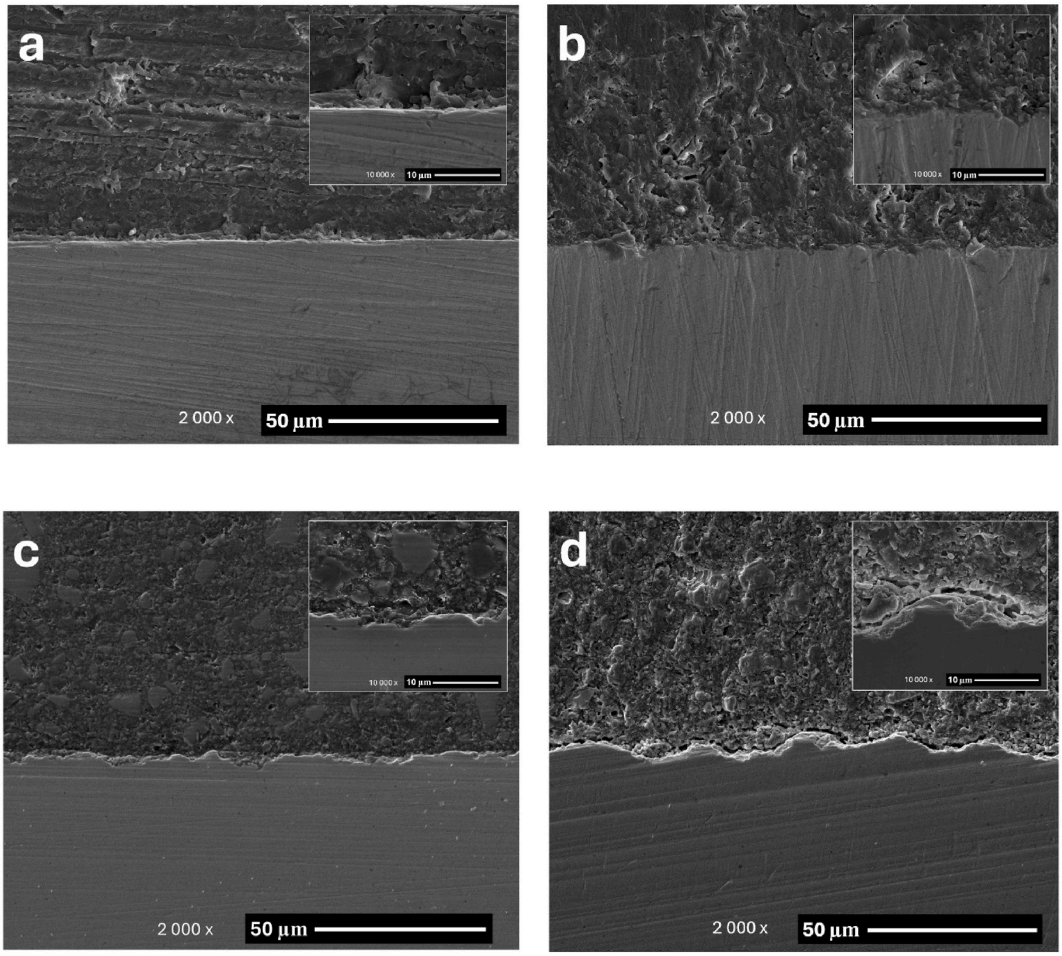

Fig. 7. SEM cross-sectional images (2000 × magnification) of resin-matrix cement bonded to zirconia with different surface treatments: (a) SB, (b) SC, (c) DLW10, and (d) DLW35. Insets at higher magnification (10,000 ×) highlight detailed interface characteristics.

Table 5
Mean shear bond strength (SBS) values (MPa ±SD) of zirconia–resin interfaces after different surface treatments under water storage and thermocycling conditions
(n = 10 per group).

Group	Water Storage (Mean ± SD)	Thermocycling (Mean ± SD)
SB	9.91 ± 3.66	5.96 ± 2.42
SC	8.49 ± 2.16	7.07 ± 2.64
DLW10	9.15 ± 1.67	7.54 ± 2.70
DLW35	8.88 ± 2.35	8.49 ± 2.02

precise material removal at the microscale with minimal surrounding damage. This makes them particularly effective for roughening the surface of zirconia ceramics. Increased surface roughness typically leads to higher surface energy, which enhances the material's capacity to bond with liquids and adhesives. This results in better wettability and stronger adhesion [39,40,69].

Among the majority of the studies that used Nd:YAG laser for zirconia surface modification to enhance bonding to resin cement only few studies reported about surface roughness while wettability and surface free energy have not been reported by any study [36]. Since surface roughness, contact angle, and surface free energy are closely linked rougher surfaces usually increase surface free energy and lower the contact angle, improving wettability and bonding. On the other hand, smoother surfaces tend to have lower surface free energy and higher contact angles, making them less ideal for bonding. Several studies have reported the correlation between surface roughness and bond strength

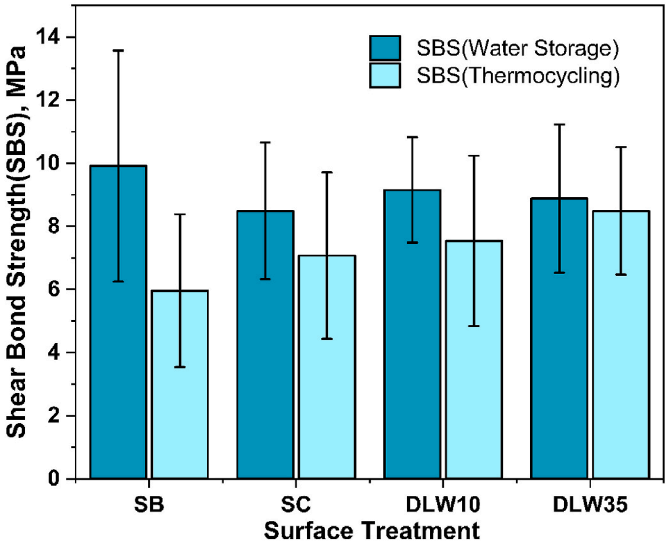

Fig. 8. Shear bond strength (SBS) values for surface treatments (DLW35, DLW10, SB, SC) under water storage (WS) and thermocycled (TC) conditions. No statistically significant differences were observed among groups or conditions ($p > 0.05$, two-way ANOVA). Thermocycling did not significantly affect SBS ($p = 0.157$).

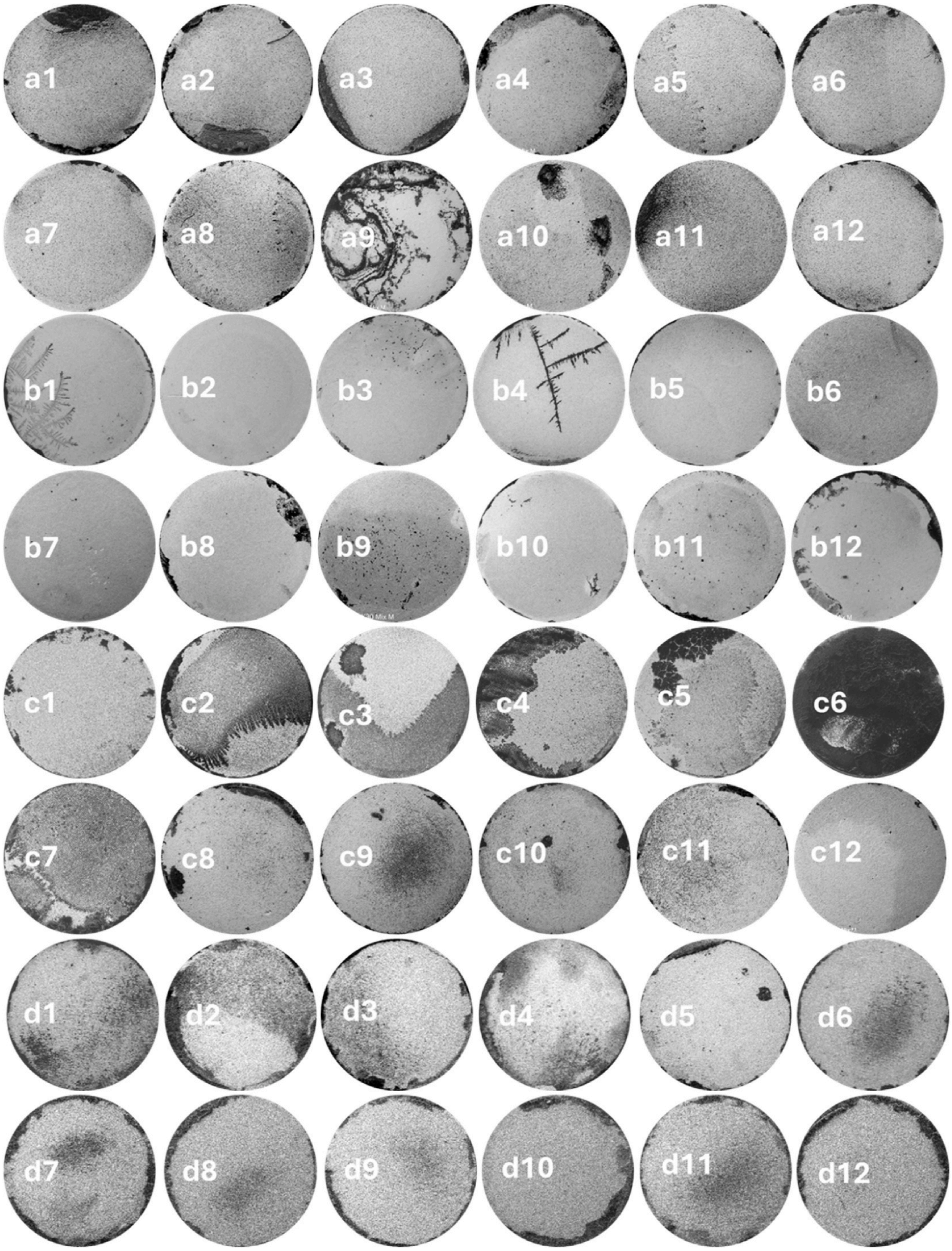

Fig. 9. – SEM images of fractured surfaces following shear bond strength tests: (a1–a12) sandblasting (SB), (b1–b12) silica coating (SC), (c1–c12) DLW10, and (d1–d12) DLW35 groups.

[39].

The sandblasting (SB) and tribochemical silica coating (SC) treatments have been extensively studied, and several drawbacks have been reported, including irregular surface roughness, microcrack formation, and significant tetragonal-to-monoclinic phase transformation in zirconia [19,40,82]. These issues, can then accelerate the low-temperature degradation and at high mechanical loads, can lead to localized stress concentrations that increase the risk of crack initiation and propagation, potentially compromising the materials structural integrity and long-term performance [83,84]. In a study conducted by Ozcan et al., a decrease of material strength was reported for such conditions, despite several contradicting results have also been reported [68,85] Similarly, several studies have reported either reduction of tetragonal phase or transformation of tetragonal to monoclinic phase after the application of SB and SC surface treatments. Okutan et al. reported 7 % increase in monoclinic content after SB treatment. Similarly, Ruja et al. reported 29 % and 26 % monoclinic content after SB and SC treatment [39,40]. In our study the monoclinic phase content was found 24 % and 32 % for SB and SC, respectively Similarly, Saade et al. also reported about increase in monoclinic phase after SB treatment and it was found to be 13 % [82].

The laser surface treatment groups (DLW10 and DLW35) exhibited increased surface roughness compared to the SB and SC groups. The surfaces in the SB and SC groups were characterized by irregular and uneven grooves, a result of the impact of sharp alumina particles. The manual application of alumina blasting introduced variability in indentation depths across the surface. Conversely, the DLW-treated surfaces demonstrated greater uniformity, with a consistent pattern of engraving achieved through laser treatment, enabling precise control over surface depth, width, and pattern via computer-aided machining and specialized software. This observation is validated by SEM images, which clearly reveal the differences in surface topography between the treated groups.

Various bond strength testing methods, including shear bond strength (SBS) and micro-shear bond strength (μSBS), are used to evaluate the adhesion between zirconia and resin-matrix cements. Among these, SBS is the most widely adopted method in dental research due to its practical relevance, ease of execution, and ability to simulate the predominant forces experienced in clinical applications. Despite its widespread use, it is recognized that the shear bond strength (SBS) test has certain limitations in accurately reflecting the true interfacial adhesion and stress states present in clinical scenarios. The SBS test was selected in this study due to its methodological simplicity, reproducibility, and extensive acceptance as a comparative metric in dental materials research, particularly for evaluating the adhesion of resin cements to zirconia substrates (ISO 29022:2013). The test enables straightforward specimen preparation, standardized testing geometry, and efficient throughput, making it possible to compare results with a broad body of existing literature.

However, it is important to acknowledge that the SBS method does not exclusively generate pure shear stresses at the adhesive interface; rather, a complex stress distribution, including tensile and compressive components, may arise during testing. These factors can influence the mode and locus of failure, potentially leading to an overestimation or underestimation of the actual interfacial strength. Additionally, SBS values may not fully represent clinical performance, as intraoral conditions and long-term loading are more complex than what is simulated in vitro.

Nevertheless, when interpreted with caution and complemented by detailed fractographic analysis, the SBS test provides valuable comparative insight into the effects of different surface treatments on resin-zirconia adhesion. For this reason, the SBS method remains a practical and widely recognized tool for initial screening of adhesive performance in restorative dental materials research [86].

Numerous studies have explored the impact of Nd:YAG laser treatment on the bond strength of zirconia ceramics, with outcomes varying widely depending on laser parameters, surface pre-treatments, and adjunctive protocols such as silanization or bioglass coatings. Several reports have shown that microsecond- or nanosecond-pulsed Nd:YAG lasers can produce bond strengths equal to or greater than those achieved by sandblasting (SB) or silica-coating (SC) particularly when higher energy densities or specific combinations with other treatments are used [36,72,79,87–90]. Conversely, multiple studies have also reported that long-pulsed or improperly optimized Nd:YAG laser treatments may yield inferior bond strengths compared to SB or SC, often due to the formation of surface flaws, increased monoclinic content, or microcracks [81,91–93]. Some investigations note that the application of silane or bioglass can improve the bond strength of laser-treated surfaces, although bioglass or silane alone may sometimes outperform the combined or laser-only treatments [37,92].

A recurring limitation in much of the literature is the frequent omission of comprehensive surface characterization, such as quantitative roughness, wettability data, or microstructural imaging, which complicates efforts to directly relate surface modifications to bond strength outcomes [19,64,65]. Where such analyses are provided, some studies report increased roughness, surface flaws, and phase transformation (notably to the monoclinic phase) after Nd:YAG laser treatment, which can variably affect adhesion [94,95]. Collectively, the literature suggests that while Nd:YAG laser treatment has significant potential to enhance zirconia–resin bonding—especially with optimized laser parameters and in synergy with other treatments—its success is highly technique-sensitive [19,64,65]. Careful surface characterization and process optimization are therefore essential for reliably translating these laboratory results into clinical improvements.

Nd:YAG laser treatment has shown potential for improving bond strength, but many studies fail to provide essential surface characterization details, such as roughness, wettability, and micrographs, which are critical for understanding the underlying mechanisms influencing bond strength. The varied outcomes also highlight the need for standardized protocols and comprehensive analyses to establish reliable conclusions.

The fracture surface analysis revealed a consistent pattern of adhesive failure across all surface-treated groups, including those subjected to abrasive treatments (SB, SC) and laser texturing (DLW10, DLW35). In all cases, the complete detachment of resin-matrix cement from the zirconia surface suggests insufficient resin infiltration into the microstructured surface, limiting the development of strong micromechanical interlocking. This lack of interpenetration likely contributed to the predominantly adhesive failure mode observed in all groups.

Despite this trend, no direct correlation could be established between the failure mode and the bond strength values, indicating that factors beyond just fracture type, such as interfacial stress distribution and polymerization shrinkage effects, may also play a role in adhesion durability. Notably, SEM micrographs of the DLW35 group revealed a distinct gap between the resin layer and the zirconia substrate, suggesting the possible presence of air entrapment during cementation or an incomplete adaptation of the resin-matrix cement to the surface features. This observation raises questions about whether highly textured surfaces, despite offering increased roughness, might also create microvoids that compromise resin adaptation and interfacial bonding. These findings emphasize the complexity of achieving optimal zirconia-cement adhesion, highlighting the need for a careful balance between surface roughness, wettability, and resin flow behavior to maximize interfacial integrity and bond strength.

In this study, all surface treatments resulted in comparable shear bond strength (SBS) values under both water storage and thermocycling conditions, demonstrating the effectiveness of both laser and airborne particle abrasion methods in enhancing the adhesion of resin-matrix cement to zirconia surfaces. The bond strengths achieved with laser-modified surfaces (DLW10, DLW35) were statistically similar to those obtained through conventional grit-blasting treatments (SB, SC), indicating that Direct Laser Writing (DLW) can serve as a viable alternative to traditional airborne particle abrasion.

Airborne particle abrasion (SB, SC) remains a widely used method for zirconia surface treatment due to its ability to increase surface area, enhance wettability, and improve micromechanical interlocking. The roughened surface produced by alumina particle blasting (SB) and tribochemical silica-coated alumina abrasion (SC, Rocatec™ system) promotes adhesion through a mechanical retention mechanism. However, the process introduces several uncontrolled variables that may compromise long-term performance. One of the primary concerns with grit-blasting techniques is the irregularity of the resulting roughness profile, which can lead to inconsistencies in bonding effectiveness. Additionally, airborne particle abrasion induces tetragonal-to-monoclinic phase transformation, particularly in SC-treated zirconia (32 % monoclinic content) and SB-treated zirconia (24 % monoclinic content). The increase in monoclinic content is known to generate residual stresses, surface microcracking, and potential long-term mechanical degradation of zirconia restorations. Moreover, particle embedding and contamination from airborne abrasion can weaken the adhesive interface by interfering with resin infiltration, further affecting bond durability.

In contrast, Direct Laser Writing (DLW) provided a controlled and uniform surface roughening approach while preserving zirconia's crystalline integrity. Unlike grit-blasting, which is applied after sintering, DLW was performed on zirconia in its green state (pre-sintering), allowing for precise material removal and the creation of tailored surface microstructures without inducing microcracks or residual stresses. This controlled process resulted in a well-defined periodic roughness pattern, minimizing surface irregularities and ensuring a more reproducible bonding interface. Despite having higher surface roughness, particularly in DLW35 ($R_a = 1.81 \pm 0.23$ μm), the absence of interfacial microcracks and contamination ensured that the resin could infiltrate the surface without structural compromise. However, fractographic analysis revealed the presence of a localized interfacial gap in one DLW35 specimen, potentially caused by air entrapment during cementation. While this observation was not widespread, it highlights the importance of optimizing resin application techniques to maximize interfacial adaptation in highly textured laser-modified surfaces.

Although laser-treated zirconia exhibited comparable bond strengths to grit-blasted groups, the uniform surface roughness and phase stability provided by DLW present distinct advantages. The SBS values for DLW10 (9.15 ± 1.67 MPa) and DLW35 (8.88 ± 2.35 MPa) were statistically similar to those of SB (9.91 ± 3.66 MPa) and SC (8.49 ± 2.16 MPa), confirming that laser-modified surfaces can achieve bond performance equivalent to airborne particle abrasion without inducing adverse phase transformation effects. Moreover, SBS values remained stable even after thermocycling, indicating that none of the surface treatments led to premature adhesive degradation over time. Beyond bond strength considerations, laser surface treatment offers distinct operational and clinical advantages. Unlike grit-blasting, which requires the use of abrasive media and controlled air pressure, DLW is a non-contact, contamination-free method that eliminates the risk of embedded particles, thereby preserving the purity of the zirconia surface. Additionally, DLW is highly adaptable, enabling the precise control of surface patterns to optimize wettability, roughness, and adhesion properties without introducing excessive material damage. Moreover, laser processing is highly scalable and automation-friendly, making it an attractive alternative for industrial applications requiring repeatability and precision. The ability to customize surface textures without inducing uncontrolled roughness or phase transformation effects further strengthens DLW's potential as a long-term replacement for conventional grit-blasting techniques in zirconia-based restorations.

The statistical analysis revealed no direct correlation between surface roughness and surface free energy (SFE) or shear bond strength (SBS), indicating that increased roughness alone does not necessarily enhance adhesion. While DLW35 exhibited the highest roughness ($R_a = 1.81 \pm 0.23$ μm), it did not result in superior wettability or bond strength compared to DLW10. Conversely, contact angles (water and diiodomethane) showed a strong negative correlation with SFE ($r = -0.99$, $p < 0.0001$), confirming that lower contact angles are associated with higher surface energy. This was evident in DLW10, which demonstrated the lowest contact angles and highest SFE, suggesting improved surface wettability. Despite these trends, SFE alone did not directly predict SBS, as all surface treatments resulted in comparable bond strength values, both in dry and thermocycled conditions. The predominant adhesive failure mode across all groups suggests that interfacial bonding mechanisms are limited, regardless of surface treatment. These findings emphasize that while optimizing surface roughness and wettability can influence SFE, achieving durable adhesion between zirconia and resin-matrix cement likely requires additional chemical or mechanical bonding strategies.

Beyond laboratory performance, the clinical adoption of Direct Laser Writing (DLW) compared to conventional airborne particle abrasion (SB or SC) depends on several practical factors. Airborne particle abrasion remains the standard in most clinical settings due to its low cost, ease of use, and minimal equipment requirements. The procedure is rapid and familiar to dental professionals, with setup and treatment times typically under a few minutes per specimen. However, airborne abrasion can introduce variability in surface quality, risk of contamination from embedded particles, and generates dust that poses health and safety concerns. In contrast, DLW offers a high degree of process control, enabling precise and reproducible surface patterning with minimal risk of contamination. While initial investment in laser equipment is significantly higher than that for air abrasion units, DLW is fully automatable and compatible with digital workflows, which may streamline laboratory processes and reduce operator-dependent variability in high-throughput or industrial settings. The operational complexity of DLW is greater, requiring specialized training and maintenance, and the time required per specimen may be longer due to setup, calibration, and laser scanning—though this can be offset by batch processing and integration with CAD/CAM systems. Thus, DLW offers distinct advantages in surface quality and process control but at higher initial cost and operational complexity, while conventional air abrasion remains cost-effective and efficient for routine clinical use. The choice between methods should therefore consider the clinical environment, caseload, and required consistency of surface modification [49,96].

This study demonstrates that DLW-treated zirconia surfaces can achieve adhesive performance comparable to airborne particle abrasion while maintaining structural integrity and phase stability. The findings reinforce the importance of surface roughness, wettability, and phase transformation control in optimizing zirconia-resin bonding. Further investigations could focus on refining DLW parameters to minimize interfacial gaps and enhance resin infiltration, ensuring even stronger adhesion in future applications.

5. Conclusions

This study demonstrates Direct Laser Writing (DLW) as an effective alternative to conventional airborne particle abrasion (SB, SC) for zirconia surface modification, achieving comparable shear bond strength (SBS) without compromising material's microstructural integrity. DLW provided uniform and reproducible surface roughness compared to the irregular and potentially contaminated grit-blasted surfaces. All treatment groups showed statistically similar SBS values under both water storage and thermocycled conditions, indicating stable long-term adhesive performance. Unlike grit-blasting methods, DLW preserved the tetragonal zirconia phase, maintaining structural integrity and avoiding detrimental phase transformations. Despite variations in roughness and wettability, adhesive failure predominated across all groups, highlighting ongoing challenges in achieving strong chemical bonding at the zirconia-resin interface. The higher surface roughness observed with DLW35 did not enhance bond strength relative to DLW10, emphasizing that excessively rough surfaces may negatively affect resin infiltration. DLW offers significant practical advantages, such as contamination-free surfaces, precise topographical control, and compatibility with automation, making it a promising method for clinical and prosthetic applications. Further research should focus on optimizing DLW parameters to enhance resin infiltration, reduce interfacial gaps, and maximize bonding performance.

CRediT authorship contribution statement

Narayan Sahoo: Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Oscar Carvalho:** Resources, Funding acquisition, Conceptualization. **Mutlu Özcan:** Writing – review & editing, Supervision. **Tan Fırat Eyüboğlu:** Writing – review & editing, Methodology. **Júlio C.M. Souza:** Writing – review & editing, Validation. **Filipe Silva:** Resources, Funding acquisition. **Bruno Henriques:** Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this work.

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