Influence of surface treatments and resin cement selection on bonding to zirconia

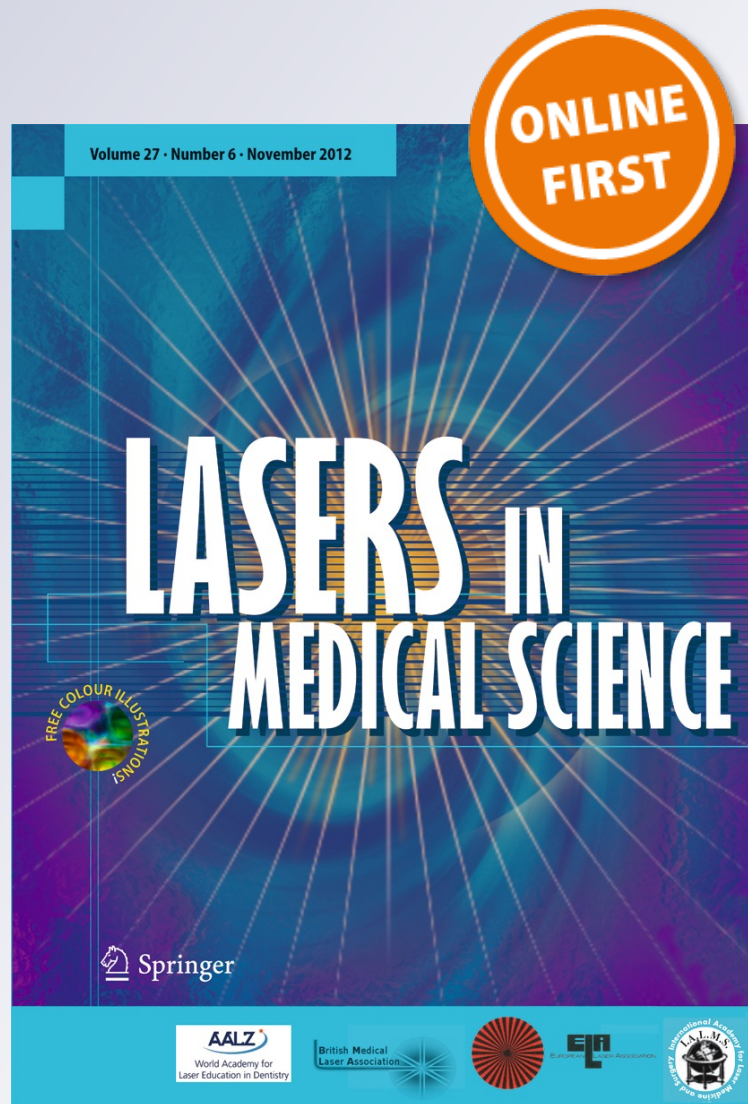## Meryem Gülce Subaşı & Özgür İnan

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ORIGINAL ARTICLE

Influence of surface treatments and resin cement selection on bonding to zirconia

Meryem Gülce Subaşı · Özgür İnan

Abstract This study aimed to evaluate the surface changes caused in zirconia by different surface treatments and the influence of the surface treatment and cement selection on bonding to zirconia under aging. Sintered zirconia specimens were divided into five groups ($n=31$) based on the surface treatment, namely, control, air abrasion, silica coating, laser and air abrasion + laser. After surface treatment, surface roughness and microscope analyses were performed on one specimen of each group. Composite cylinders were then bonded to conditioned ceramics using RelyX U100 (RXU), Clearfil Esthetic Cement (CEC) and Panavia F (PF) ($n=10$). After 24 h, the bonded specimens were subjected to thermal cycling (6,000 times), and then, a shear bond strength test was conducted. The roughness values were analysed using Kruskal–Wallis and Mann–Whitney U tests, and the bond strengths were analysed by two-way analysis of variance and Duncan's test. The relationship between the roughness and the bond strength was determined by Spearman's correlation analysis. Specimens subjected to surface treatments were rougher than the control specimen ($p<0.000$). However, there were no significant differences between the air abrasion and air abrasion + laser groups and the silica coating and laser groups. Specimens treated with laser showed lower bond strengths irrespective of the resin cement used. CEC and/or PF showed higher bond strengths than RXU for each surface treatment group.

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No significant relationship was observed between the roughness and the bond strength. The results of this study showed that all the surface treatments, except for laser irradiation, were suitable for treating zirconia ceramics. Cement selection was found to be more important than surface treatment, and phosphate monomer-containing cements were suitable for cementing zirconia.

Introduction

In recent years, high-strength zirconium oxide ceramics have attracted increasing attention for use in oral rehabilitation [1–3] owing to their good mechanical and aesthetic properties [4–6]. Computer-aided design/computer-aided manufacturing techniques can be used for milling dental restorations from presinterised [7] or with the final dimensions from fully sinterised yttrium-stabilised tetragonal zirconia (Y-TZP) blocks [8].

The use of different types of cement (conventional, glass ionomer, self-adhesive and resin) has been proposed for luting zirconia. Among these, adhesive resin cements are preferred owing to its better retention and marginal fit [9, 10]. Phosphate monomer-based (MDP) [11–14] and self-adhesive resin cements are recommended for zirconia-based restorations [15–18]. The phosphate ester group in MDP-based resin cements [19, 20] and the multifunctional acid methacrylates in self-adhesive resin cements [12, 21, 22] have been reported to interact with a porcelain surface and facilitate adhesion.

To achieve reliable adhesion to ceramics, it is typically necessary to carry out a surface pre-treatment. Roughened ceramic surfaces may allow resin cements to flow into the

microretentions and create a stronger micromechanical interlock [11, 15].

Unlike conventional dental ceramics, high-strength ceramics such as Y-TZP have high crystalline content and comprise many particles without a glass phase at the crystalline border [23]. Therefore, these ceramics are not suitable for acid etching [11, 24–26]. In this light, other surface treatment methods such as air abrasion with aluminium oxide (Al_2O_3) [11, 21, 27–29] or silica coating with silica-modified Al_2O_3 particles [12, 16, 21, 27] are frequently employed for Y-TZP restorations. In addition to currently used ceramic conditioning methods, the erbium:yttrium–aluminium–garnet (Er:YAG) laser is used in clinical dentistry applications such as the removal of carious dentin [30], cavity preparation [31] and surface treatment of indirect restorations [32–34]. Macroscopic and microscopic irregularities remaining on the surface after Er:YAG laser application may play an important role in bonding [31].

The long-term success of zirconia-based restorations depends on the preparation technique of the internal surfaces of ceramics prior to cementation, cement properties and bond strength between the cement and the ceramic [15, 24, 27]. Numerous studies [11, 12, 15, 16, 21, 24, 27–29, 34] have already focused on these issues; however, there remains controversy over the applicable surface treatments and type of luting agents for adhesion to Y-TZP materials.

This study aimed to evaluate the influence of surface treatments on the surface morphology and roughness of zirconia and to determine the bond strength of different resin cements to zirconia after different surface treatments and aging. The following hypotheses were tested: (1) surface treatment does not affect the surface morphology and roughness of zirconia and (2) neither surface treatment nor resin cement selection influences the bond strength at the cement–ceramic interface.

Materials and methods

A single type of Y-TZP ceramic material (VITA In-Ceram YZ for inLab®; VITA Zahnfabrik, Bad Säckingen, Germany, batch number 24430) was used in this study. This ceramic consists of 92 % ZrO_2, 5 % Y_2O_3, <3 % HfO_2 and <1 % Al_2O_3 and SiO_2 by weight. One hundred fifty-five sintered and polished ceramic specimens (12×11.2×2.4 mm) were divided into five groups (*n*=31) according to the surface treatment used.

Group 1, Control: No surface conditioning procedure was used for this group.

Group 2, Air abrasion: The ceramic surfaces were air-abraded with 110-μm Al_2O_3 particles (Korox; Bego, Bremen, Germany) from a distance of approximately 10 mm at a pressure of 3 bar for 15 s.

Group 3, Silica coating: Ceramic surfaces were air-abraded using an intraoral device (Cojet; 3M ESPE AG, Seefeld, Germany) filled with 30-μm silica-coated Al_2O_3 particles (Cojet Sand; 3M ESPE AG, Seefeld, Germany) from a distance of approximately 10 mm at a pressure of 3 bar for 15 s.

Group 4, Laser treatment: An Er:YAG laser (AT Fidelis Er:YAG; Fotona, Ljubljana, Slovenia) with a wavelength of 2,940 nm was irradiated on the ceramics using a special handpiece (R14-C). The laser cylindrical sapphire optical fibre (diameter, 1.3 mm) was placed perpendicular to the surface at a distance of 1 mm, and the restrained ceramic area was scanned with water irrigation and air cooling for 15 s. The laser parameters were as follows: energy, 400 mJ; pulse rate, 10 Hz; power, 4 W; and MSP mode pulse width, 100 μs.

Group 5, Air abrasion + laser treatment: Specimens were air-abraded, and then, they were irradiated using the Er:YAG laser. Air abrasion was carried out because it is a conventional laboratory procedure. The same parameters as those used in air abrasion and Er:YAG laser treatment were used for this group.

After the surface treatments, the samples were ultrasonically cleaned in 99 % acetone for 5 min and then in distilled water for another 5 min to remove excess sand particles from the surface, in a manner similar to the process given by Yang et al. [35].

Surface roughness evaluation

After the conditioning procedure, the average roughness (Ra, unit: micrometre) of each specimen was measured using a profilometer (Mitutoyo Surftest 402; Mitutoyo Corporation, Minatoku, Japan) with a cut-off value of 0.8 mm and measuring length of 4 mm. Prior to the measurement, the profilometer was calibrated against a reference block with an Ra of 2.792 μm. Five measurements were performed at different locations near the centre of the specimen, and their average was used to obtain the average roughness of each specimen.

Microscope analyses

A sample from each surface treatment group, the roughness of which was equal or most similar to that group's average roughness value, was selected and used for these analyses. Atomic force microscope (AFM) and scanning electron microscope (SEM) analyses were used to qualitatively observe the morphological changes on the zirconia surfaces after different surface treatments.

For AFM analysis, a Multimode Nanoscope 3D AFM (Veeco Metrology Group, CA, USA) was used and digital images were recorded in air. Scans were performed in the

intermittent contact mode using a 0.5–2-Ωcm Si tip (at 50 μm) oscillating at a frequency of 297,354 kHz. For each specimen, a 40×40-μm digital image was recorded using a slow scan rate (1 Hz). Then, the images were evaluated using special software (Nanoscope (R) IIIa Version 5.31R1).

For the SEM analysis, a JEOL 6335F SEM (Japan Electron Optics Ltd., Tokyo, Japan) was used with an accelerating voltage of 15 kV. For this analysis, the ceramic specimens were first sputter-coated with gold–palladium particles using Quorum Technologies Polaron SC7640 (Newhaven, East Sussex, UK) for 15 s to a thickness of 30–40 Å. Then, images of each specimen were captured at ×700 magnification.

After the roughness and microscope analyses, each group of pretreated ceramic specimens was divided into three subgroups (n=10) according to the selected resin cements: (1) RelyX U100 (RXU; 3M ESPE AG, Seefeld, Germany); (2) Clearfil Esthetic Cement (CEC; Kuraray, Okayama, Japan); and (3) Panavia F (PF; Kuraray, Okayama, Japan). A piece of polyethylene tape (Scotch Magic Tape Invisible; 3M, France) with a 5-mm-diameter circular hole was positioned on the ceramic surface to control the bonding area. Then, composite resin cylinders (5 mm (Ø)×2 mm (height)) (Filtek Z250; 3M ESPE, St. Paul, MN, USA) were prepared in a mould and light-cured (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) for 40 s. They were then bonded to the conditioned ceramic surfaces by the cements according to the manufacturer's instructions. The chemical composition and application mode of the resin cements are described in Table 1.

Luting was carried out under a constant load of 3 kg in a universal testing machine (TSTM 02500; Elista Ltd. Şti., İstanbul, Türkiye). The seating force was applied for the first 5 min to allow the material to set in self-curing modality. Oxygen-inhibiting gel (Oxyguard II; Kuraray, Okayama, Japan) was applied on the free surfaces, and light irradiation was performed for an additional 40 s on each side of the specimens (for a total of 200 s) to ensure optimal polymerisation. The light intensity was verified to not be lower than 600 mW/cm^2 using a radiometer (Demetron; Kerr, USA). The bonded specimens were rinsed and stored in distilled water for 24 h at 37 °C. Subsequently, all specimens were subjected to thermal cycling (custom-made thermal cycling machine; Nova Ticaret, Konya, Türkiye) for 6,000 cycles between 5 and 55 °C with a transfer time of 2 s and dwell time of 30 s. The shear bond strength was tested using a universal testing machine at a crosshead speed of 0.5 mm/min until failure occurred, and the bond strength values were recorded in units of megapascals.

The fractured surfaces of all tested specimens were analysed under a stereomicroscope (Olympus SZ40; Olympus Optical Co., Tokyo, Japan) at ×10 magnification. The fracture patterns were classified as adhesive (A) (between the cement and the ceramic), cohesive (C) (within the cement or ceramic), or mixed (M) (adhesive and cohesive fractures occurred simultaneously).

Statistical analysis

The data for the surface roughness were not normally distributed (Kolmogorov–Smirnov test, p=0.01). Therefore, all levels were compared by nonparametric analyses using the Kruskal–Wallis and Mann–Whitney U tests. The bond strength values were analysed by two-way analysis of variance (ANOVA). Duncan's tests were used to compare the bond strength values of the surface treatment and cement groups. Spearman correlation analysis was used to assess the relationship between the surface roughness and the bond strength values of the specimens. All statistical analyses were performed with a significance level of p=0.05.

Results

Roughness analysis

Table 2 lists the mean, median, standard deviation, minimum and maximum values of the surface roughness (Ra, unit: micrometre) for the five groups and the results of the Kruskal–Wallis and Mann–Whitney U tests. According to the Mann–Whitney U test, all the surface-treated specimens were rougher than the control group (p<0.000). However, there were no significant differences between the air abrasion and air abrasion + laser or silica and laser groups.

Microscope analyses

AFM and SEM images of ceramic specimens with different surface treatments are shown in Figs. 1 and 2, respectively. The resulting surface topographies for the control and laser groups were similar, and both of these showed smoother surface profiles than the other groups (Figs. 1a, d and 2a, d). The air abrasion, silica and air abrasion + laser groups showed changes in the surface texture with the formation of microretentive grooves (Figs. 1b, c, e and 2b, c, e).

Bond strength and failure analyses

According to two-way ANOVA, the cement type (F=35.87; p<0.000) and surface treatment (F=20.70; p<0.000) significantly affected the shear bond strength to zirconia. Interactions between the cement/surface treatment factors (F=2.48; p<0.05) were also significant. Table 3 summarises the mean and standard deviations of the bond strengths and Duncan's test results for the tested groups.

Regardless of the surface treatment performed on the ceramic, the bond strength of PF was significantly higher than that of RXU. In specimens cemented with RXU, those treated with laser exhibited the lowest bond strength and those treated with air abrasion exhibited higher bond

Table 1 Component, chemical composition and application mode of the resin cements tested in the study

Material	Component	Composition[a]	Application
RelyX U100 (TR)	Base	Glass powder, silanated silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator	Mix cement, apply, self-cure (5 min) and light cure (40 s)
Batch no. 400952	Catalyst	Methacrylated phosphoric esters, dimethacrylates, acetate, stabilisers, self-cure initiators, light-cure initiators	
Clearfil Esthetic Cement (clear)	Clearfil ceramic primer	3-MPS, MDP, ethanol	Apply primer on ceramic, wait for 5 min and air dry
Batch no. 10ADB	Paste A	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, colloidal silica	Mix cement, apply, self-cure (5 min) and light cure (40 s)
	Paste B	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass fillers, colloidal silica, catalyzers, dl-camphoroquinone, pigments	
Panavia F2.0 (light)	Clearfil Porcelain Bond Activator	Bisphenol A polyethoxydimethacrylate, 3-MPS	Mix Clearfil Porcelain Bond Activator and Clearfil Se Bond Primer and apply on ceramic, wait for 5 min and air dry
Batch no. 404AA	Clearfil Se Bond Primer	HEMA, water, hydrophilic dimethacrylate, 10-MDP, camphoroquinone, tertiary amine	Mix cement, apply, self-cure (5 min) and light cure (40 s)
Batch no. 072BA	Paste A	MDP, dimethacrylate, silanated silica, chemical and photoinitiators	
	Paste B	Dimethacrylates, sodium aromatic sulfinate, accelerator, sodiumfluoride, silanated barium glass	

[a] According to the information provided by the manufacturers

TR translucent, *Bis-GMA* bis-phenol A diglycidylmethacrylate, *TEGDMA* triethylene glycol dimethacrylate, *MDP* 10-methacryloxydecyl dihydrogen phosphate, *3-MPS* 3-methacryloyloxypropyltrimethoxysilane, *HEMA* hydroxyethylmethacrylate

strengths than did the control and laser-treated specimens ($p < 0.05$). Furthermore, the control, silica and air abrasion + laser specimens showed similar bond strength values, as did the air abrasion, silica and air abrasion + laser specimens. In specimens cemented with CEC, the control specimen and those treated with air abrasion, silica and laser showed similar bond strengths with no significant differences. However, specimens treated with air abrasion + laser exhibited bond strengths that were statistically higher than those of specimens treated by silica and laser but similar to those of the control specimen and the specimens treated by air abrasion. In specimens cemented with PF, those treated with air abrasion, silica and air abrasion + laser exhibited higher bond strengths than did the control specimens and those treated by laser ($p < 0.05$); nonetheless, the former and latter groups of specimens showed no significant difference in the bond strength.

The relationships between the surface roughness (Ra, unit: in micrometre) and bond strength values (in megapascal) were compared for each surface treatment and resin cement, and no significant correlation was determined ($p > 0.05$).

The failure modes of debonded specimens are summarised in Table 4. In general, the laser group mostly showed adhesive failures, and all other surface treatment groups showed mixed failures. With regard to the cements, very few cohesive failures, mostly mixed failures, and mostly adhesive failures were observed in the CEC group, MDP-containing cement groups (CEC and PF) and RXU group, respectively.

Table 2 Results of the statistical analysis of surface roughness values (Ra, unit: micrometre)

Groups	N	Mean	Median	Standard deviation	Min	Max	Kruskal–Wallis test	
							Chi-square value	*p* value
Control	30	0.22	0.21 c	0.04	0.15	0.32		
Air abrasion	30	0.74	0.74 a	0.13	0.51	0.98		
Silica	30	0.35	0.29 b	0.13	0.22	0.67	111.392	0.000
Laser	30	0.34	0.30 b	0.14	0.14	0.70		
Air abrasion + laser	30	0.75	0.73 a	0.15	0.50	1.22		

The same letters in the same column indicate no significant differences according to Mann–Whitney *U* test ($p > 0.05$)
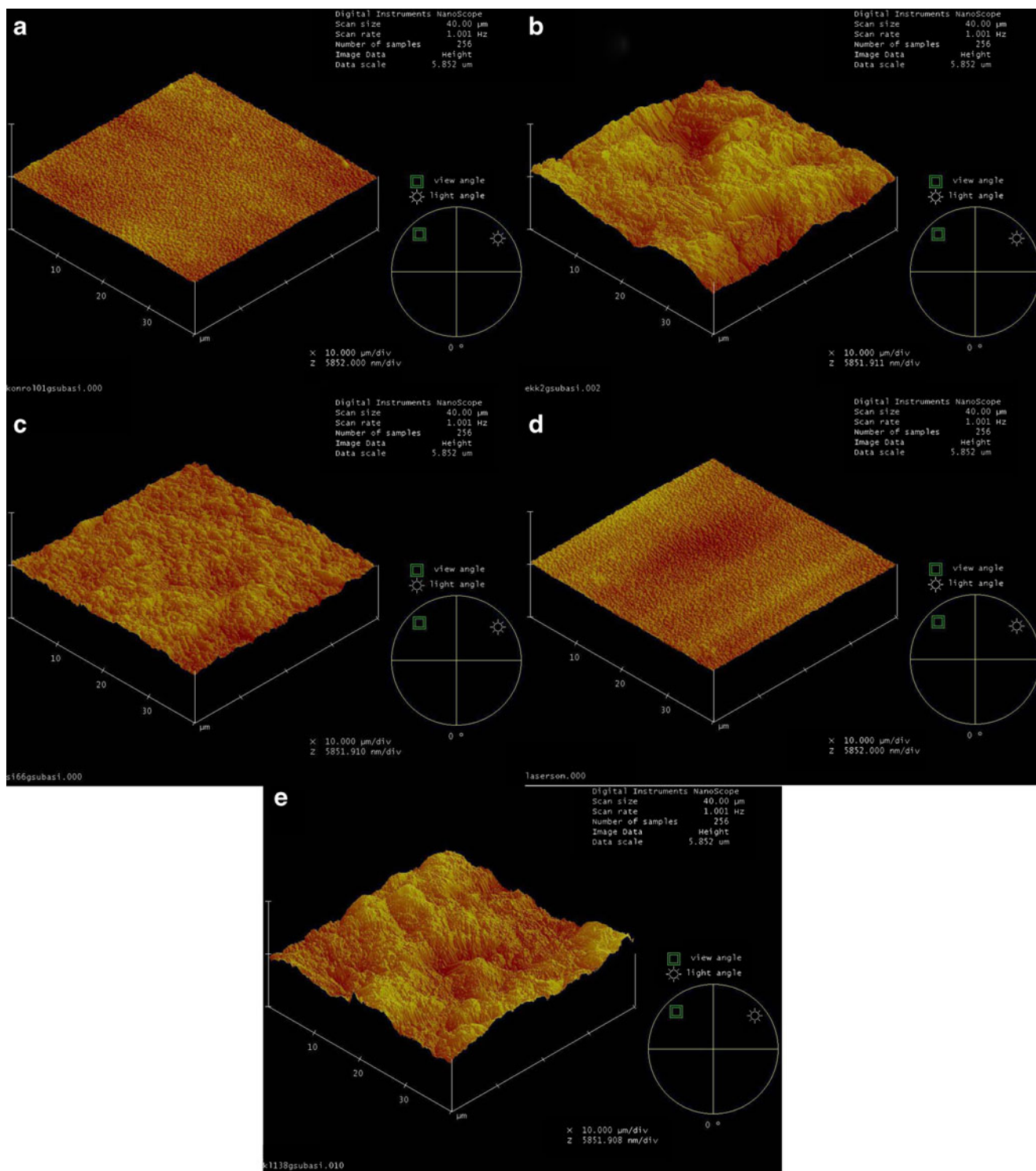
Fig. 1 AFM (40×40 μm) images of zirconia surfaces. **a** Control (untreated). **b** Air abrasion with 110 μm Al_2O_3 particles. **c** Silica coating with 30 μm silica-coated Al_2O_3 particles. **d** Er:YAG laser irradiation with 400 mJ. **e** Air abrasion + laser

Discussion

This study evaluated the surface changes caused in zirconia by different surface treatments and the influence of the surface treatment and resin cement on bonding to zirconia under aging. All the treatments were used to achieve micromechanical retention on the ceramic surface. The particle sizes used for the air abrasion and silica coating were selected based on

Fig. 2 SEM (×700 magnification) images of zirconia surfaces. **a** Control (untreated). **b** Air abrasion with 110 μm Al$_2$O$_3$ particles. **c** Silica coating with 30 μm silica-coated Al$_2$O$_3$ particles. **d** Er:YAG laser irradiation with 400 mJ. **e** Air abrasion + laser
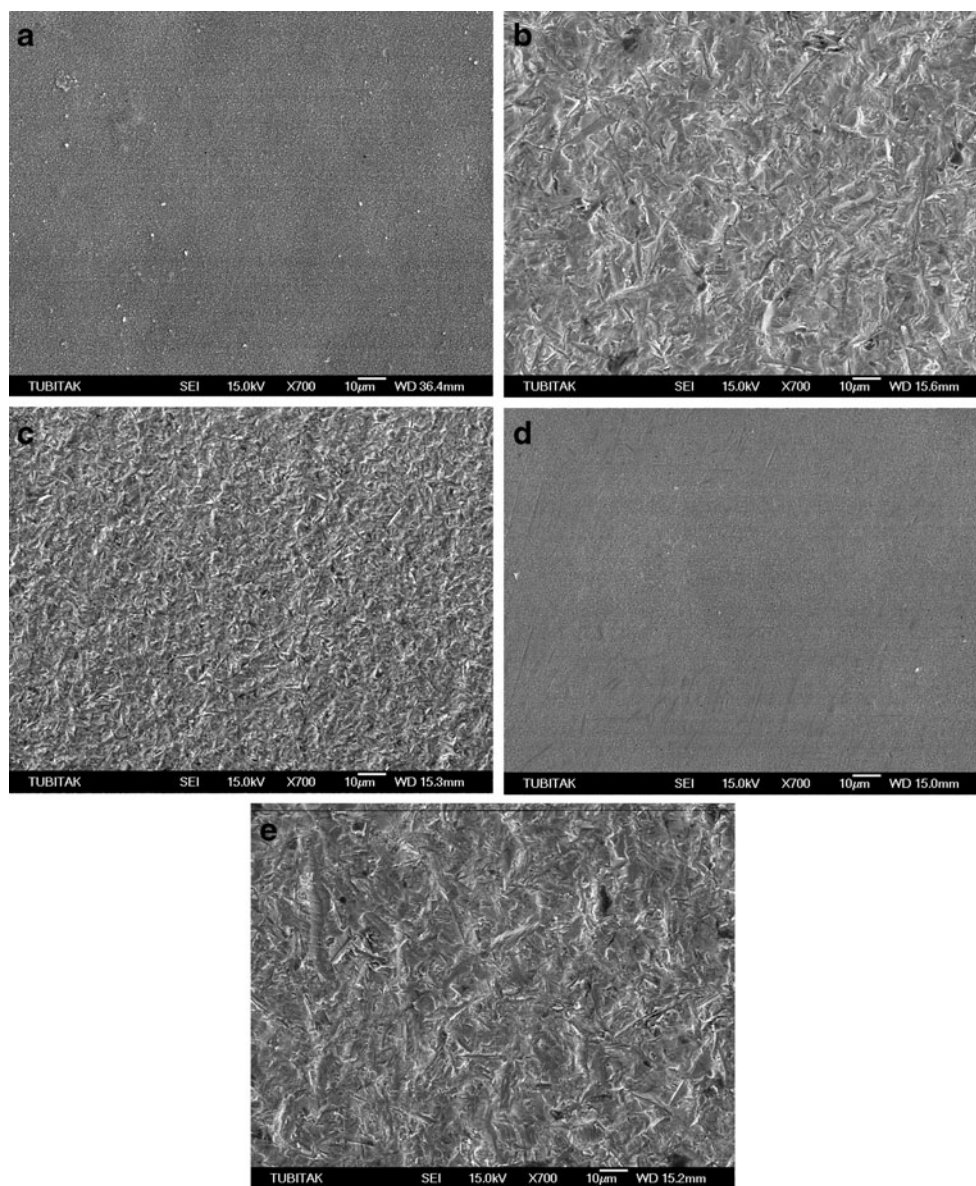
previous studies [36, 37], and the laser parameters used were determined after a preliminary study. Five sintered zirconia specimens were subjected to five different Er:YAG laser energies (200, 250, 300, 400 and 600 mJ) with the same parameters (contact mode, 10 Hz; 100 MPS; 15 s). SEM and surface roughness analyses were performed. The effect of 400-mJ Er:

YAG laser irradiation on the ceramic surface was observed to be more pronounced than that of 200-, 250- and 300-mJ irradiation, and no ceramic loss was observed. Therefore, the laser energy was selected as 400 mJ.

For the evaluation of the resin cement and ceramic interface, pretreated zirconia surfaces were cemented on standard

Table 3 Results of the statistical analysis of shear bond strength values (in megapascal) of groups

Same numbers in the same row and same letters in the same column indicate no significant differences according to Duncan's test ($p > 0.05$)

Surface treatment	RXU		CEC		PF	
	Mean	SD	Mean	SD	Mean	SD
Control	2.43 b, 1	0.62	3.52 ab, 3	0.39	2.95 a, 2	0.23
Air abrasion	3.07 c, 1	0.55	3.47 ab, 12	1.04	4.00 b, 2	0.26
Silica	2.59 bc, 1	0.35	3.09 a, 12	0.95	3.74 b, 2	0.89
Laser	1.76 a, 1	0.37	2.79 a, 2	0.64	2.48 a, 2	0.38
Air abrasion + laser	2.72 bc, 1	0.77	4.11 b, 2	0.68	4.18 b, 2	0.58

Table 4 Percentage distribution of failure mode

	RXU			CEC			PF		
	A	C	M	A	C	M	A	C	M
Control	90	–	10	30	10	60	–	–	100
Air abrasion	60	–	40	10	30	60	10	–	90
Silica	50	–	50	70	–	30	–	–	100
Laser	100	–	–	60	–	40	40	–	60
Air abrasion + laser	40	–	60	–	–	100	–	–	100

A adhesive; *C* cohesive, *M* mixed

resin composite cylinders instead of tooth structures owing to the uniform structure of the composite resin. This prevented the results from being misinterpreted owing to the microstructural variables of the tooth structure [14, 18].

In this study, the thermal cycling method was performed as an aging procedure because the chemical, thermal and mechanical factors in the mouth can affect the bond strength between the resin cement and the ceramic [11, 12, 15, 16, 19, 21, 27, 28, 38]. A standardised electronic thermal cycling device was used according to the ISO/TS 11405 [39] recommendations. As in previous studies [16, 27, 38], short-term thermal cycling (6,000 times) was performed; this corresponded to a duration of approximately 7 months [40].

The findings of this study required the rejection of both initial hypotheses because variations occurred in the surface topography and roughness after surface treatments. Furthermore, surface treatment and cement selection affected the bond strength between the resin cement and the ceramic. However, the selection of the luting cement seemed to be a more relevant factor with regard to bonding to the zirconium oxide ceramic.

The tested surface-treated specimens were rougher than the control group ($p < 0.000$). Microscopic analyses revealed changes in the surface topography in the air abrasion, silica and air abrasion + laser groups and the laser group with the formation of microretentive grooves and pits, respectively. The 400-mJ Er:YAG laser seemed to have a minimal impact on zirconia; this might be attributed to less absorption of laser energy by zirconia ceramics owing to the fact that they are water-free materials.

The bond strength of CEC in the control, laser and air abrasion + laser groups and that of PF in all surface treatment groups were both higher than RXU ($p < 0.05$). This was because the MDP contents of both CEC and PF and the adhesiveness of MDP-containing cements increased the bond strength, revealing the capability of acidic functional monomers reacting with the substrate [21, 28]. Moreover, among the cement groups, very few cohesive failures, mostly mixed failures, and mostly adhesive failures were observed in the CEC group, MDP-containing cement groups (CEC and PF) and RXU group. Mixed and cohesive failures are clinically preferable to adhesive failures because adhesive failures are usually associated with low bond strength values [41]. The failure mode also supported the bond strength results.

In studies of zirconia ceramics [11, 42] cemented with PF, the use of an MDP-based coupling agent after air abrasion or silica coating has been reported to yield higher bond strengths than the use of an MDP-based primer without surface treatments. As in these studies, the bond strength values of specimens that were treated by air abrasion, silica and air abrasion + laser and cemented with PF were found to be higher than those of the control specimens and those treated by laser ($p < 0.05$). Thus, the MDP primer in PF may chemically bond better to aluminium oxide and silica than to zirconium oxide. Moreover, for PF, micromechanical and chemical retention may play an important role in the bonding to zirconia ceramics.

Previous studies [18, 35] reported that after aging, the air abrasion group showed higher bond strength values than the control group when cemented with a self-adhesive resin cement. Similarly, specimens cemented with RXU, such as the air abrasion group, exhibited higher bond strengths than did the control and laser groups ($p < 0.05$). Although specimens cemented with RXU mostly showed adhesive failures, cement residuals were mainly observed when RXU was luted to specimens treated with air abrasion and air abrasion + laser, which might be due to the micromechanical retention created by these procedures.

Each of the three cements was evaluated in terms of the different surface treatments; the laser group was found to have lower bond strength values than the other groups, and adhesive failures were mostly observed only in this group. During laser irradiation, microexplosions could form debris that might adhere to the melted ceramic surfaces. We believe that such a layer might strongly bond to the resin cement; on the other hand, it might be poorly attached to the infra-layer of the surface, causing lower bond strengths.

The results of this study indicated that surface treatments affected the surface changes, and the bond strength between the resin cement and the zirconia depended on the selection of the cement rather than the surface treatment. The effects of surface treatments on the mechanical properties of zirconia ceramics and the long-term durability of the bonds of

these resin cements to pretreated zirconia ceramics will be evaluated in further studies.

Conclusions

Within the limitations of this study, the following conclusions were drawn:

1. All the treated ceramic specimens had rougher surfaces than the untreated ones ($p<0.000$). Microscope analyses showed that air abrasion, silica and air abrasion + laser treatment and laser treatment led to changes in the surface texture through the formation of microretentive grooves and small pits, respectively.
2. All surface treatment methods except laser treatment were suitable for use on the zirconia surface before cementation.
3. Surface treatment or cement selection could affect the bond strength between the resin cement and the zirconia. However, cement selection was more important than surface treatment, and phosphate monomer-containing cements (CEC and PF) were suitable for cementing zirconia.

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