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ORIGINAL ARTICLE

Effect of different surface treatments on roughness of IPS Empress 2 ceramic

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Abstract The aim of this study was to evaluate the influence of different surface treatments (air abrasion, acid etching, laser irradiation) on the surface roughness of a lithium-disilicate-based core ceramic. A total of 40 discs of lithium disilicate-based core ceramic (IPS Empress 2; Ivoclar Vivadent, Schaan, Liechtenstein) were prepared (10 mm in diameter and 1 mm in thickness) according to the manufacturer's instructions. Specimens were divided into four groups (n=10), and the following treatments were applied: air abrasion with alumina particles (50 µm), acid etching with 5% hydrofluoric acid, Nd:YAG laser irradiation (1 mm distance, 100 mJ, 20 Hz, 2 W) and Er:YAG laser irradiation (1 mm distance, 500 mJ, 20 Hz, 10 W). Following determination of surface roughness (R_a) by profilometry, specimens were examined with atomic force microscopy. The data were analysed by one-way analysis of variance (ANOVA) and Tukey HSD test (α=0.05). One-way ANOVA indicated that surface roughness following air abrasion was significantly different from the surface roughness following laser irradiation and acid etching (P<0.001). The Tukey HSD test indicated that the air abrasion group had a significantly higher mean value of roughness (P<0.05) than the other groups. No significant difference was found between the acid etching and laser irradiation (both Er:YAG and Nd:YAG) groups (P>0.05). Air abrasion increased surface roughness of lithium disilicate-based core ceramic surfaces more effectively than acid-etching and laser irradiation.

Keywords AFM · Surface treatment · Ceramic · Laser · Roughness

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Introduction

The increasing demand for metal-free fixed partial dentures has fuelled the development of all-ceramic materials with optimized mechanical properties [1]. The development of these tooth-coloured indirect restorative materials has also contributed to significant changes in the field of luting cements [2, 3]. The adhesive technology used for fixing current tooth-coloured all-ceramic restorations involves the use of resin-based luting materials [2, 3].

The advantages of adhesion of all-ceramic crowns and bridges are considered to include improved retention, better marginal adaptation and fracture resistance [4]. The internal surface of the ceramic restoration must be prepared to optimize micromechanical retention of the cement into the ceramic microroughness. Surface treatment of porcelain increases the surface area and creates microporosities on the surface, enhancing the potential for mechanical retention of the cement [5, 6]. Different surface treatment methods have been proposed to provide roughness and promote micro-mechanical retention [7, 8].

The lithium-disilicate glass ceramic IPS Empress 2 has a high crystalline content and exhibits significantly higher bond strengths than IPS Empress independent of surface conditioning [9, 10]. It seems that the ceramic microstructure has a significant influence on the fracture resistance of the composite ceramic adhesion zone [9, 10].

Table 1 Surface roughness values of each treatment group (means±SD)

Treatment group	Roughness value (mean±SD)
Air abrasion	0.62±0.17
Acid etching	0.33±0.18
Nd:YAG laser	0.25±0.01
Er:YAG laser	0.27±0.10

Air abrasion is abrasion with airborne particles of aluminium oxide using a chair-side device. This procedure increases the surface area and surface energy for adhesion of resin cements and promotes micromechanical retention. It also decreases surface tension, thereby enabling optimal wetting of silanes or adhesive media [11]. Acid etching with solutions of hydrofluoric (HF) acid or ammonium bifluoride can achieve proper surface texture and roughness [12–15]. The glassy matrix is selectively removed, and crystalline structures are exposed [14, 15]. Various applications of dental lasers on dental materials have also been proposed for surface modification, such as forming a glazed surface layer on ceramics, the removal of resin composite filling materials, laser welding of ceramics and metal alloys, including titanium, and increasing the corrosion resistance of metal alloys [16].

Scanning and transmission electron microscopes and atomic force microscopes are the most commonly used tools to observe surface details. These techniques require extensive processing of samples and observation in vacuum, which alters the surface structure. These techniques offers many advantages such as direct observation and high resolution, and they can operate in a variety of environments. Here we consider atomic force microscopy (AFM), which is becoming an accepted tool for surface roughness characterization (quantification) [17].

The aim of this study was to examine the effect of different surface treatments (air abrasion, Nd:YAG laser irradiation, Er:YAG laser irradiation and acid etching) on the roughness of IPS Empress 2 ceramic. The null hypothesis was that Nd:YAG and Er:YAG laser treatments would not increase surface roughness more than the other surface treatment methods.

Materials and methods

Specimen preparation

Forty lithium-disilicate-based all-ceramic discs (10 mm in diameter and 1 mm in thickness) were waxed (YETI Dentalprodukte, Engen, Germany), sprued, and then pressed after investment. All procedures were performed with IPS Empress 2 materials (Ivoclar, Schaan, Liechtenstein), following the manufacturer's recommendations. No glaze was applied to the ceramic surface of the discs. The bonding surface of each disc was polished using silicon carbide paper (grit 300, 400 and 600) on a rotating metallographic polishing device (Isomet 1000; Buehler, Lake Bluff, IL) under water cooling. The surfaces were cleaned with ethanol and dried carefully in air before surface treatment. After the finishing procedures, the discs were subjected to ultrasonic treatment (Biosonic UC 50; Coltene Whaledent, Cuyahoga Falls, OH) in distilled water to remove any surface residues and dried. Thereafter, the ceramic discs were randomly divided into four groups ($n=10$), according to the surface treatments applied:

Group I Air abrasion: Air abrasion with 50-µm aluminium oxide particles (Korox; Bego, Bremen, Germany) at a pressure of 2.8 bar, from a distance of 10 mm, perpendicular to the treated surface for 20 s.

Group II Acid etching: Treatment surfaces of ceramic discs were etched with 5% HF acid (IPS Ceramic Etching Gel; Ivoclar Vivadent, Schaan, Liechtenstein) for 20 s. The gel was rinsed off with water for 20 s, then dried with oil-free compressed air.

Group III Nd:YAG laser irradiation: Nd:YAG laser (DEKA M.E.L.A., Calenzano, Italy) was used for irradiation of the ceramic surfaces. The laser optical fibre (320 µm in diameter) was placed perpendicular to the ceramic surface at 1 mm distance and the whole ceramic area was scanned with water and air cooling using an adjustable air and water spray. The laser parameters used were: 100 mJ (pulse energy), 20 Hz (pulses per second), 2 W (power setting), 141.54 J/cm^2 (energy density), 150 µs (pulse length).

Group IV Er:YAG laser irradiation: Nd:YAG laser (Twinlight dental laser system; Fotona, Ljubljana, Slovenia) was used for irradiation of the

Table 2 Results of the Tukey HSD test (P values) comparing the roughness values between the treatment groups

	Air abrasion	Acid etching	Nd:YAG laser	Er:YAG laser
Air abrasion	–	0.000	0.000	0.000
Acid etching		–	0.414	0.623
Nd:YAG laser			–	0.986
Er:YAG laser				–

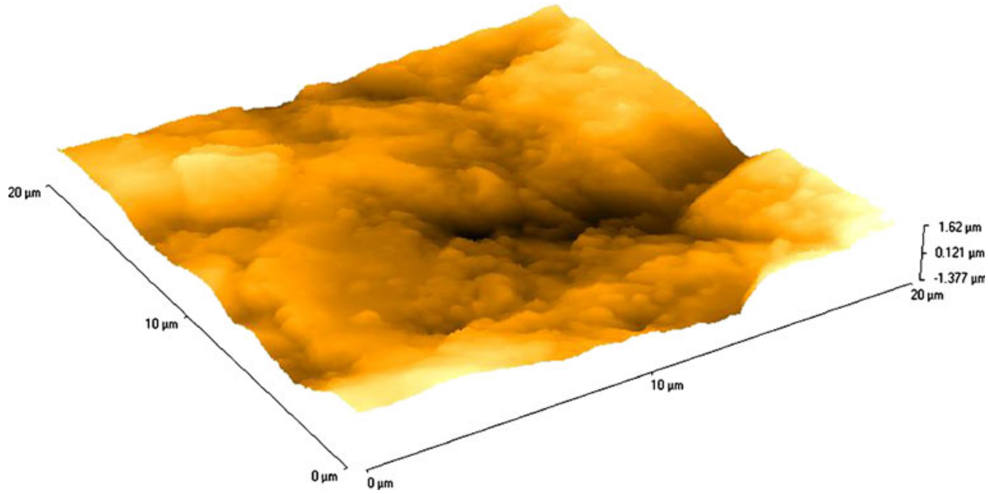

Fig. 1 AFM image of an IPS Empress 2 surface treated with air abrasion

ceramic surfaces. The laser optical fibre (400 µm in diameter) was placed perpendicular to the ceramic surface at 1 mm distance and the whole ceramic area was scanned with water and air cooling using an adjustable air and water spray. The laser parameters used were: 500 mJ (pulse energy), 20 Hz (pulses per second), 10 W (power setting), 75 µs (pulse length).

Surface roughness evaluation

The average surface roughness (R_a, microns) of the treated specimens was measured by profilometry (Surftest-402 surface roughness tester; Mitutoyo Corporation, Tokyo, Japan). Three traces were recorded for each specimen at three different locations in each direction (parallel, perpendicular and oblique) giving nine tracings per sample. The average of these nine mean surface roughness measurements was used as the score for each sample. The scores were entered into a spreadsheet (Excel; Microsoft, Seattle, WA) for calculation of descriptive statistics. The data were analysed by one-way analysis of variance (ANOVA) and the Tukey HSD test (SPSS/PC, version 10.0; SPSS, Chicago, IL) for pair-wise comparisons among the groups ($\alpha=0.05$).

AFM evaluation

One additional specimen from each group was evaluated under an atomic force microscope (Veeco Dicaliber, Santa Barbara, CA). Digital images were taken in air. A 0.01–0.025 Ω cm phosphorus-doped (n-doped) silicon tip (50 µm) was used in tapping mode. Changes in vertical position provided the height of the images, registered as bright and dark regions. The tip-sample "tap" was maintained constant through a constant oscillation amplitude (set-point amplitude). Five 20 µm×20 µm digital images were acquired for each surface and recorded with a slow scan rate (1 Hz).

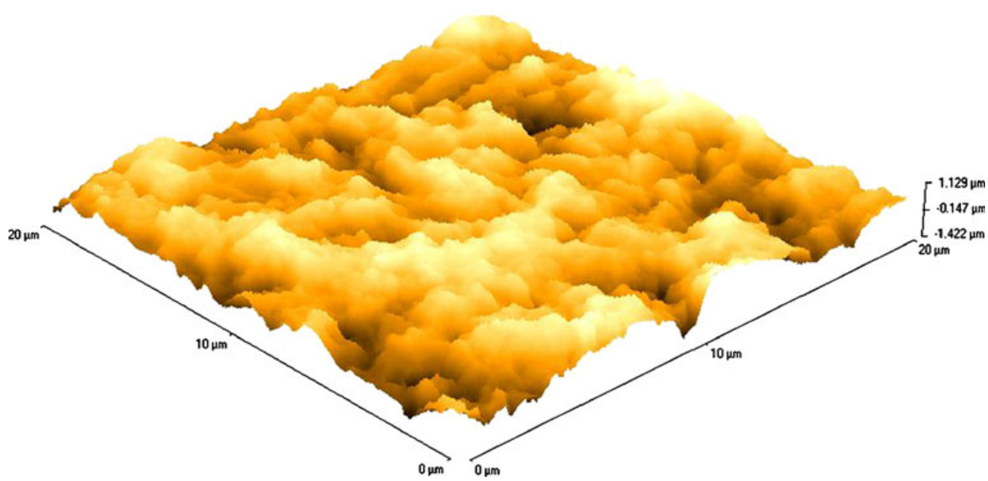

Fig. 2 AFM image of an IPS Empress 2 surface treated with HF acid etching

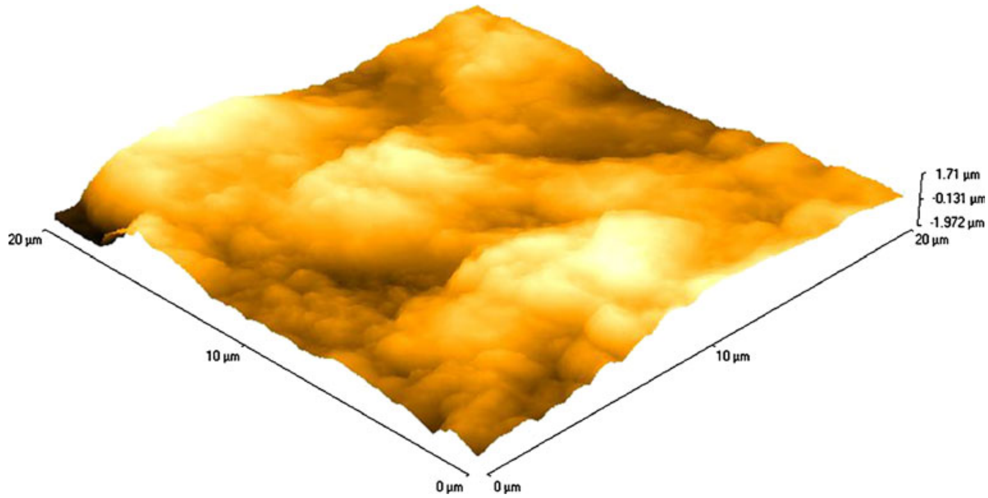

Fig. 3 AFM image of an IPS Empress 2 surface treated with Nd:YAG laser irradiation

Results

The mean surface roughness values of the four surface treatment groups are presented in (Table 1) . One-way ANOVA indicated that surface roughness following air abrasion was significantly different from the roughness following laser irradiation and acid etching ($P<0.001$). The results of the Tukey HSD test comparing the roughness values between the groups are presented in (Table 2). The air abrasion group had a significantly higher mean roughness value ($P<0.05$, Tukey HSD test) than the other groups (Table 2). No significant difference was found between the acid etching and laser irradiation (Er:YAG and Nd:YAG) groups ($P>0.05$).

AFM images of the ceramic surfaces treated with the different techniques are presented in Figs. 1, 2, 3 and 4. Air-abraded surfaces showed a non-uniform pattern with distinct sharp projections dotted with pores. Laser-irradiated (Er:YAG, Nd:YAG) surfaces showed moderate irregularity with peaks and valleys, and showed less roughness than air-abraded surfaces. Acid-etched surfaces were slightly rougher than laser-irradiated surfaces, and the peaks were higher and wider than those of the laser-irradiated surfaces.

Discussion

The strength of the bond between resin and a ceramic surface relies on micromechanical interlocking and chemical bonding, which require roughening and cleaning of the surface for adequate activation [7, 12–15, 18–20]. Several pretreatment procedures have been reported and have been used clinically to produce a micromechanical retentive ceramic surface [6, 9, 21, 22]. This study demonstrated an alternative ceramic etching pattern produced by laser treatment in comparison to conventional HF acid conditioning [23]. AFM images showed a moderately irregular surface with peaks and valleys. It was hypothesized that these irregularities would enhance the mechanical retention between the resin composite and the porcelain. The null hypothesis was accepted: Er:YAG and Nd:YAG laser treatments did not increase surface roughness more than the other surface treatment methods (air abrasion, acid etching). The study indicated that laser irradiation (Er:YAG, Nd:YAG) and acid etching produce similar surface roughness.

Micromechanical bonding was achieved by roughening the ceramic surface with 50-μm grain sized pure aluminium oxide particles. Surface roughening increases

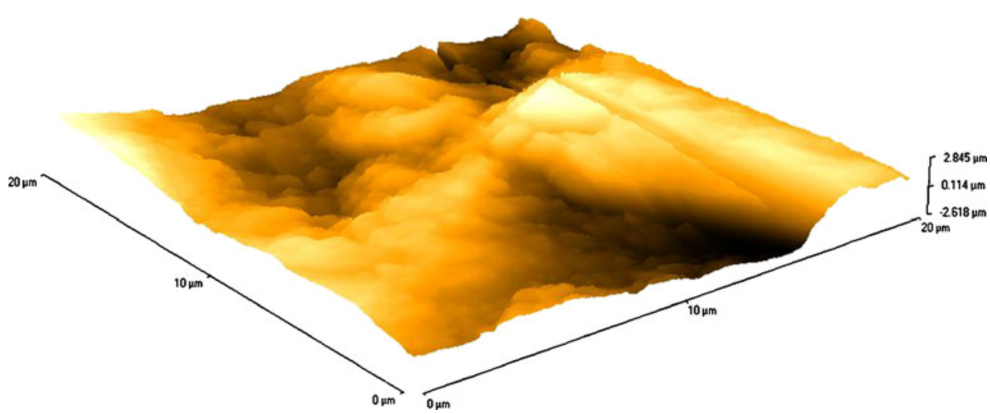

Fig. 4 AFM image of an IPS Empress 2 surface treated with Er:YAG laser irradiation

the total bonding area and also the wettability of the ceramic with the composite resin. In a study by al Edris et al. [24], the effectiveness of a chemical etchant was noticeably different between glazed and air-abraded dental ceramics. Lacy et al. [25] showed that air abrasion provides sufficient bond strength. Also in the present study, air-abraded specimens showed the highest surface roughness, and AFM images of air-abraded specimens showed a non-uniform surface with distinct sharp projections dotted with pores.

Etching with HF acid solution can produce proper surface texture and roughness [12–15]. HF acid roughens the surface by dissolving the crystalline and glassy phases of the ceramic surface [23, 26–31]. Estafan et al. [32] reported that etching of a pressable leucite-reinforced feldspathic porcelain IPS Empress ceramic with 9.6 % HF acid for 60 s provided good bond strength when using a microhybrid composite resin. The lithium-disilicate glass-ceramic IPS Empress 2 has a high crystalline content and exhibits significantly higher bond strengths than IPS Empress independent of surface conditioning [9, 10]. It seems that the ceramic microstructure has a significant influence on the fracture resistance of the composite–ceramic adhesion zone [9, 10].

The Er:YAG laser acts on the dental substrate by thermomechanical ablation. The water in the dental tissues is vaporized causing its expansion followed by micro-explosions that eject both organic and inorganic tissue particles, providing a surface with open dentinal tubules and no smear layer [33]. The extent of the superficial changes on the ceramic surface depends on the energy density of the laser radiation as well as on the type of irradiated ceramic [26]. Shiu et al. [26] observed that Er:YAG laser irradiation of a feldspathic ceramic surface at 1 W output power (100 mJ per pulse at 10 Hz) did not lead to adequate roughening of the surface because of the composition of the ceramic and its reflectance. Although in this study, a higher output power of the Er:YAG laser (500 mJ per pulse at 20 Hz) than that used by Shiu and colleagues was used, it was found that laser-treated surfaces were not significantly different from HF acid-treated surfaces. Gökçe et al. [21] reported that the shear bond strength after laser treatment at 300 mJ was higher than after treatment at 600–900 mJ. According to them, the reason for the low bond strengths observed after laser treatment at high power settings could have been related to the observed heat-damaged layer. This layer might be poorly attached to the infralayers of the substrate, while the outermost layer of the substrate remained still strongly bonded to the silane and luting agents.

Nd:YAG laser irradiation has been proposed for modifying the surface of ceramics by forming a glazed surface layer [16]. Li et al. [23] found that the pulsed Nd:YAG laser with appropriate energy parameters could replace etching in the pretreatment of porcelain surfaces for bonding with composite resin. Osorio et al. [6] evaluated the effect of different surface treatments (sandblasting, Nd:YAG laser irradiation, Rocatec universal bonding system) on the roughness of In-Ceram Alumina. They selected an output power of 2 W (100 mJ per pulse at 15 Hz) for laser irradiation. In their AFM study, similar topographic images were found for all surface treatments. Akyıl et al. [31] reported that HF acid etching provides a higher shear bond strength than Nd:YAG laser (1 W, 100 mJ per pulse at 10 Hz) etching. In the present study, there were no significant differences between laser irradiation and HF acid etching.

Conclusion

Within the limitations of the present in vitro study, the following conclusions could be drawn:

1. Air abrasion created rougher surfaces than the other surface treatment methods (Er:YAG and Nd:YAG laser irradiation, Acid etching; $P<0.05$).
2. There were no significant differences in surface roughness following Nd:YAG laser irradiation, Er:YAG laser irradiation and HF acid etching ($P<0.05$).
3. AFM images of laser-irradiated (Er:YAG, Nd:YAG) surfaces showed moderately irregular surfaces with peaks and valleys.

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