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Original Article

The effect of ytterbium-doped fiber laser on titanium fused to ceramic

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Abstract

Aim: The aim of our study is to compare the application of fiber lasers with aluminum oxide sandblasting and CoJet, which is one of the other surface roughening methods used for the first time in this study. Investigating the effect of titanium-porcelain bond strength is another aim of this study.
Methodology: In total, 96 samples of titanium discs (6 mm in diameter and 3 mm in height) were prepared in a CAD/CAM device. Samples were divided into six groups (n: 10), including the control group, according to the applied surface treatments: one-way (horizontal) scanning with a Yb fiber laser, two-way (horizontal-vertical) scanning with a Yb fiber laser, three-way (horizontal-vertical-hypotenuse) scanning with a Yb fiber laser, CoJet, sandblasting, and the control group. After using the different surface treatments on the titanium discs, scanning electron microscope (SEM) imaging, a wettability test, and a profilometer test were applied to the samples in each group. After the surface treatments were applied to the titanium discs, including those in the control group, low heat porcelain (VITA Titanium Porcelain), which was 4 mm in diameter and 3 mm in height, was applied according to the manufacturer's instructions. Afterwards, porcelain samples prepared with titanium inlays were placed in acrylic blocks and kept in 37°C (±1°C) distilled water for 24 hours. Shear tests were applied to the samples embedded in prepared acrylic blocks, and the results were evaluated.
Results: In the statistical evaluation of the obtained data, one-way analysis of variance (one-way ANOVA) was used to compare the statistical differences of the mean values of the bond strength between titanium and porcelain. In our study, Tukey's HSD and Dunnett's multiple comparison statistical tests were used to determine the differences among the groups.
Conclusion: According to the results of our study, the highest average bond strength values were obtained from sandblasting and three-way fiber laser roughness. As a result, no statistically significant difference was found among the groups. Although Yb fiber laser application was not found to have a statistically significant effect on titanium porcelain bond strength, this method could be useful for titanium porcelain bonding.

Keywords: titanium, ytterbium-doped fiber laser, bond strength, surface roughening, porcelain

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Introduction

Strong bonding to porcelain is essential for the long-term success of metal ceramic restorations (1). Titanium has been used in metal-ceramic restorations because of its several advantages (2-5), such as exceptional biocompatibility (2-5, 6), excellent corrosion resistance (2-5, 7), and high strength to low density (4.5 g/cm) ratio (2-5).

The bonding of titanium dental ceramics is a different and sensitive process. The union of titanium to ceramic in metal and ceramic fixed partial dentures remains problematic because of the lack of a strong bond between ceramic and metal substructures (3). The surface structure and composition of titanium are crucial to establishing a good bond (2). Titanium has a high melting point and a high gas affinity of oxygen, hydrogen, nitrogen, and carbon gases (8). In the presence of oxygen, an oxide layer is formed that adheres to the titanium surface. While this oxygen layer confers corrosion resistance, it decreases the bond strength (9) at the metal ceramic interface considerably (4). Various pure metals and ceramics have been used to coat titanium surfaces to prevent oxidation during firing. Laser welding is another currently available option.

Laser technology appears to be a noncompeting technology, as it keeps replacing other conventional surgical procedures in dentistry due to its precision level, accuracy, and productivity. Laser irradiation is thought to be an alternative method to increase surface roughness and improve adhesion between ceramics and metals (10–14). The laser types used for surface treatment are the Er:YAG laser, the Nd:YAG laser, the CO_2 laser, and the ytterbium-doped fiber laser (YbPL) (15–17). Erbium and ytterbium lasers are widely used in biomedical applications. While the Yb^{3+} ion transitions offer potentially high pumping efficiency due to their small quantum defects, ytterbium lasers operate near 1 µm and offer small nonradiative losses, low heating, almost 80% conversion efficiency and 25% wall-plug efficiency (18). To prevent damage to the surface, laser settings, such as pulse, power, and duration, are of great importance.

The purpose of this study was to evaluate and compare the effects of a YbPL on the bond strength of a titanium surface to a ceramic surface and on the surface roughness of titanium to those of other surface treatment methods, such as sandblasting with Al_2O_3 or 110 µm silica-coated alumina (the CoJet system). The hypotheses tested were that YbPL would not influence the surface roughness of titanium and the shear bond strength (SBS) of titanium to ceramic.

Materials and Methods

For the study, 96 titanium disc specimens (12 for SEM examination, 12 for wettability, and 12 for profilometer examination) that were 6 mm in diameter and 3 mm in height were cut from Kera Ti 5-Disc Cad-Cam Titanium blocks (ZirkonZahn, Steger, Italy) using CAD/CAM technology (Yenemak, Kayseri, Turkey).

Laser irradiation

The titanium disc surfaces were irradiated using a Yb-doped fiber-based nanosecond pulsed laser with 20W power output (Vision, Neukirchen, Germany). The irradiation was carried out with YbPL at 1,064 nm with frequencies of 25 kHz and 100 kHz, 1 mJ pulse energy, and a pulse duration of 100 ns (an ultrashort pulse). The laser beam was directed over the zirconia disc surface in a noncontact mode at a working distance of 18.0 mm, with the laser focusing on the zirconia disc surfaces via vertical, horizontal, and hypotenuse scanning (Fig. 1). The spot size of the laser device was less than 50 µm, and it had an air-cooling system.

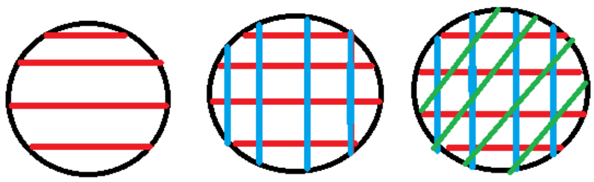

Figure 1. Different direction and number of fiber laser scanning shapes. a) Horizontal unidirectional roughening with fiber laser, b) Horizontal and vertical two-way roughening with fiber laser, c) Horizontal, vertical and hypotenuse three-way roughening with fiber laser

Experimental groups

All titanium disc specimens were randomly divided into six groups, with each group composed of 16-disc specimens according to the surface treatment methods applied to them. The control group had no surface treatment.

Group 1 (G1): Disc surfaces were sandblasted with one scan (horizontal) of YbPL irradiation at 825W power output and 100 kHz.

Group 2 (G2): Disc surfaces were sandblasted with two scans (horizontal and vertical) of YbPL irradiation at 25W power output and 100 kHz.

Group 3 (G3): Disc surfaces were sandblasted with three scans (horizontal, vertical, and hypotenuse) of YbPL irradiation at 25W power output and 100 kHz.

Group 4 (G4): disc surfaces were sandblasted with 30-µm silica-coated alumina (CoJet System, 3M ESPE) at a constant pressure of 280 kPa for 20 s/cm2 and at a perpendicular distance of 10 mm.

Group 5 (G5): disc surfaces were sandblasted with Al_2O_3 particles (110 µm) for 15 s at 0.41–0.68 MPa and a distance of 10 mm.

Group 6 (G6): Control group, no surface treatment was done.

Six specimens were randomly selected from each group to determine the effects of different surface treatments using SEM (n=2), wettability (n=2), and profilometer (n=2) analysis.

Applying the ceramic with the layering technique on titanium discs

Once surface treatments of titanium samples were finished, they were treated with the ultrasonic cleaner (Sharpertek Pontiac, Michigan, USA) for 300 seconds before getting air-dried.

Factoring in the shrinkage rates calculated for porcelain in layering applications, a duplicating silicone mold (Duosil D, Shera, Germany) of 4 mm height and 3 mm diameter upon firing was prepared to use as a superstructure ceramic mold, and its dimensions were proofed with a caliper.

Titanium disc samples were cleaned under pressurized hot steam for 10 seconds before applying the superstructure porcelain. Titanium porcelain powder and porcelain liquid (Vita, Titankeramik, Vita, Germany) were mixed as per manufacturer instructions to obtain a porcelain clay of suitable consistency, and the dentine porcelain was molded with condensation technique to minimize porosity. Samples were carefully removed from the mold and fired according to the procedure, as per the manufacturer's instructions.

Applying shear strength

Bond strength of the samples was tested with the micro-tensile/shear device (Esetron Mekatronik, Ankara, Turkey) in the Research Laboratory of Afyon Kocatepe University. To prepare titanium ceramic samples for use with the test device, they were fixed in the acrylic cast in rigid PVC molds with a diameter of 3.5 cm and height of 2.5 cm (Imicryl, Konya, Turkey) and stored in 37±1°C distilled water for 24 hours. The cutting device of the test apparatus was positioned so that its tip formed a 90° angle to the superstructure ceramic surface. To complete the shear test, a load of 0.5 mm/min approach velocity was applied on the titanium ceramic interface until the ceramic was separated from the titanium disc (Fig.2). The value of the force at the point of separation was recorded in Newtons. To determine the amount of load per unit area, the force applied was divided into the area of the superstructure ceramic, and the shear strength was recorded in MPa's.

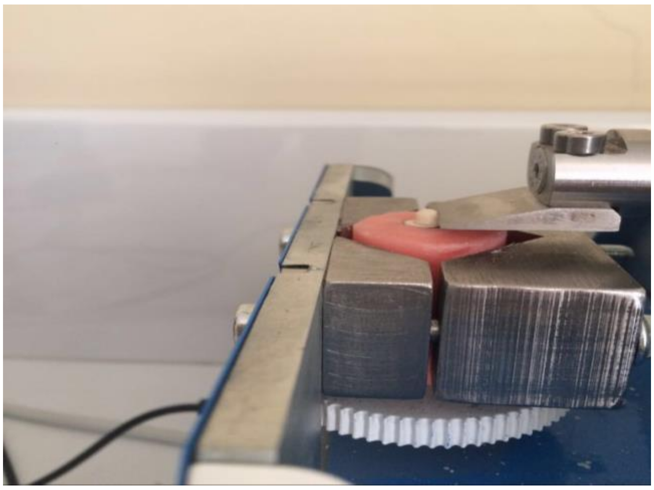

Figure 2. Applying shear force on the micro tensile/shear device.

Examining the Failure Types

Following the shear test, the separation patterns of all samples were examined with x10 magnification under a stereomicroscope (Z16 APO, Leica, Wetzlar, Germany).

This examination showed three types of separation:

1. Adhesive separation; where the ceramic of the superstructure was completely separated from titanium disc.
2. Cohesive separation; where the superstructure ceramic was completely broken.
3. Combined separation; where both types (adhesive + cohesive) were observed.

Evaluation of surface wettability

Titanium specimens were measured with a goniometer to examine the effects of laser irradiation and surface roughness methods on wetting and surface energy (Attension Theta, Stockholm, Sweden). The contact angles (θ) of the distilled water on the untreated control group and surface roughness group's surfaces specimens were detected in atmospheric condition at 25°C using a sessile drop measure machine (First Ten Angstroms, Inc., VA, USA). The specimens were cleaned with acetone before the measurement, rinsed with distilled water, and dried to eliminate contaminant layers. Contact angles were measured based on a previous study, and the mean value of each group was calculated. A lower contact angle value might indicate the best wettability for a titanium surface (14).

Evaluation of surface roughness

The surface roughness of the specimens was measured with a non-contact profilometer (NANOVEA 3D, CA). The cutoff value was set at 0.8 mm. Each titanium sample was tested three times, and the mean values of these measurements were adopted as indicated by the corresponding specimen. The Ra values were measured, and the mean value of each group was calculated. Higher Ra values might indicate a rougher surface.

Examination with a scanning electron microscope

A scanning electron microscope (SEM) (S-3400N; Hitachi High-Technologies Corporation, Tokyo, Japan) was used to observe the surface treatment effects. The analysis procedures were carried out after gold sputtering with 250, 1000, 2500, 5000, and 10,000 magnifications.

Statistical analysis

Analysis of the data was carried out with SPSS software version 22.0 (IBM Corp., Armonk, NY, USA). In this study, descriptive statistics values consisted of; mean, standard deviation, standard error, minimum and maximum values. The Kolmogorov-Smirnow test was used to assess whether the data agreed with the normal distribution assumption, and their homogeneity was assessed by the Levene's test.

Moreover, the Independent-samples t-test was used to determine the difference between two mean values in dependent groups, One Way Anova (ANOVA) was used to determine the differences between mean values where more than two groups were present, and Tukey-HSD and Dunnett multiple comparison statistical analysis tests were used to determine the differences between groups.

A 95% confidence interval was used in statistical assessments. Descriptive statistics and analyses were performed with the free software package 'R version 3.2.3 (2015-12-10), Copyright © 2015 The R Foundation for Statistical Computing.' Results where $p<0.05$ were considered statistically significant.

Results

Although the bond strength values were obtained as Newtons, the strength values were converted to MPa for this study. The mean bond strength and standard deviation (SD) of the groups were measured and listed in Table 1. The higher mean bond strength was 37,035 MPa for Al_2O_3 group (G5) and 36,668 MPa for three ways fiber laser group (G3). The lower mean bond strength was 24,930 MPa for the control group (G6).

The bond strengths of the laser groups were; 32,097 MPa for one scan (G1), 28,055 MPa for two scans (G2), and 36,668 MPa for three scans (G3). It was observed that the mean shear bond strength increased according to the number of laser scans.

According to the ANOVA test, there is no statistically significant difference between all groups ($p<0,05$).

Examining the failure types

Classification of failure types was done according to the failure types on titanium disc surfaces. For all groups, no adhesive failures were seen. %91,3 ratio of combine type failure and %8,7 ratio of cohesive failure were seen. Combined failure (Fig.3 a, b, c) and cohesive failure (Fig.3d) views obtained from the samples are given below.

a b c d

Figure 3. a, b, c. Combined failure d. Cohesive failure

Evaluation of surface wettability

According to the contact angle analysis, the highest angle value was observed in the horizontal single scanning group with fiber laser (141°) (Fig. 4a), and the lowest angle value was observed in the control group (96°) (Fig 4b). Wettability is inversely proportional to the contact angle. Thus, when the surface wettability between the groups was compared, the most hydrophobic group was the fiber laser and horizontal single scanning group with the highest contact angle, and the most hydrophilic group was the control group with the lowest contact angle.

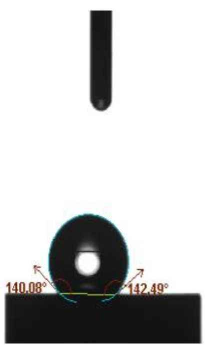

Figure 4a. Surface angle for G1

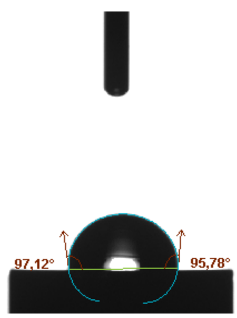

Figure 4b. Surface angle for G6

Evaluation of surface roughness of the discs

With the examination, the highest surface roughness result was seen with a value of 2.74 µm in the one scan with fiber laser (G1) group (Fig 5a). The lowest surface roughness result was seen in the control group sample with a value of 0.404 µm (Fig. 5b). Surface roughness results were higher in the samples whose surface was roughened with fiber laser compared to all other groups.

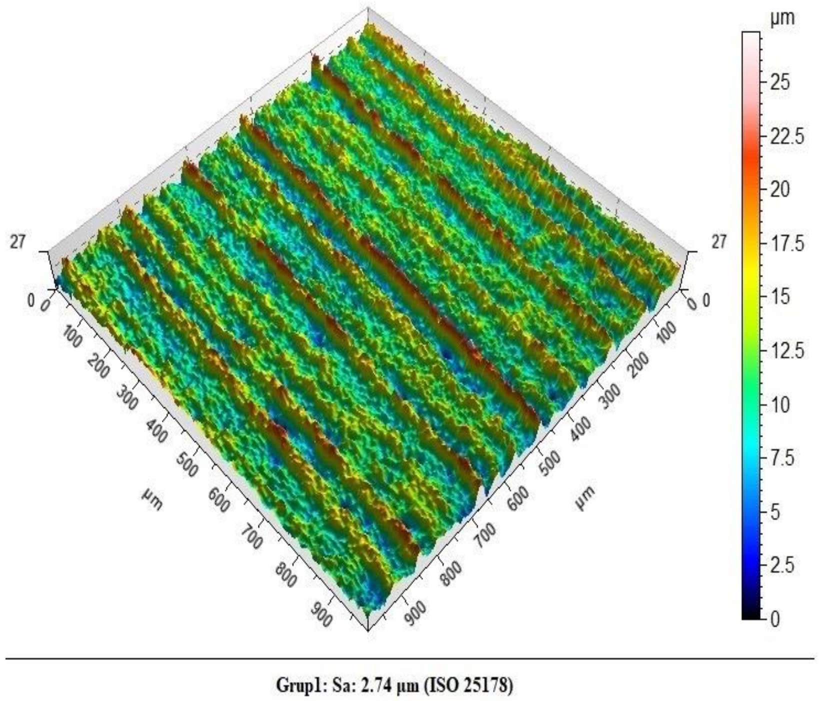

Figure 5a. Surface roughness for G1

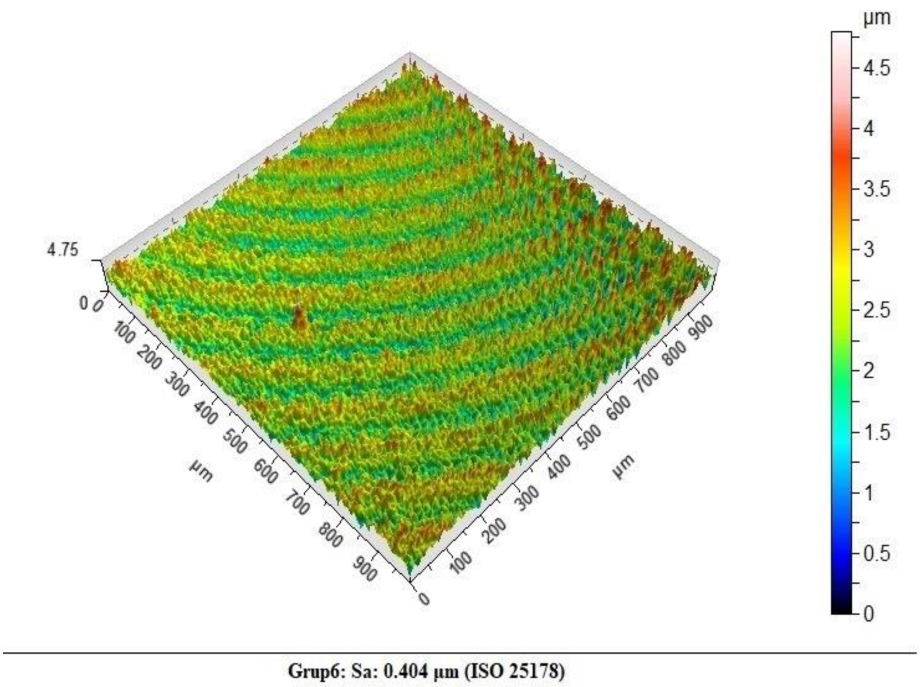

Figure 5b. Surface roughness for G1

Evaluation of SEM analysis of the disc surfaces

In the SEM analysis examination, a uniform surface is observed in the control group samples (G6), while completely flat ring floors are observed when compared to the other groups. In the scanning groups made with fiber laser, it is seen that mossy fluctuations and protrusions occur after scanning. It is seen that there is an increase in the mossy fluctuations that occur on the ground as the number of hatching increases, and therefore the surface formations increase in proportion to the number of hatches. The highest fluctuation, namely surface formations, is among the fiber laser groups; was seen in the three-way fiber laser scanning group (G3). On the other hand, a weaker surface image was observed in the Cojet group (G4), and a micro-cracked appearance was obtained in the form of light glass shards, which can be seen more clearly at 1000x and 2000x magnifications. In the samples whose surface was shaped by sandblasting, there was an appearance close to the crater at 500x magnification, while more slit-like sharp formations were seen at 1000x and 2000x magnifications.

Discussion

The search for new prosthetic treatment materials is driven by the fact that the metal in the metal-ceramic systems used in dentistry can cause allergies, the general lack of biocompatibility in the metal infrastructure, the corrosion of the metal, and the excess specific gravity of the metal infrastructure in cases such as full-arch prosthetic restorations (1). Titanium is a tissue-friendly material with superior biocompatibility and mechanical properties. In addition, it is lightweight with high corrosion resistance. However, the titanium-porcelain bond appears to be weaker than the traditional metal-porcelain bond (19).

The weak connection between titanium and porcelain is attributed to two factors: the incompatible thermal expansion coefficients of titanium and porcelain (19-21) and the excessive affinity of titanium for oxygen, which affects the properties and formation of the oxide layer (19, 22-24). As the temperature increases, the affinity of titanium for oxygen increases, and an excessive oxide layer thus forms on the titanium (19, 23, 25). The thickness of this oxide layer lowers the bond strength between the titanium and the porcelain (19, 22, 26), and this causes a major problem in the titanium-porcelain connection. Namely, this layer is responsible for most adhesive fractures between the titanium substructure and the porcelain (20, 22, 25, 27, 28). Kimura et al. (26) claimed that porcelain furnace temperatures should ideally be below 883˚C to form a minimal oxide layer.

Titanium and titanium alloys can be produced using various techniques in dentistry practice (27, 29, 30). Haag and Nilner (30) reported that the bond strength of titanium can be insufficient due to the characteristics of the oxide layer formed on the surface and produced by the casting technique. Moreover, Pang et al. (27) asserted that there is no significant difference between the titanium produced by casting and milled titanium in terms of porcelain adhesion.

Following the research, surface roughness that increases the surface area of the titanium substructure is necessary to obtain a sufficient connection because it affects the bond strength between titanium and porcelain (21, 28, 31-33). Sandblasting with aluminum oxide (Al_2O_3) is one such surface roughening process, and it both reduces surface tension and increases surface area (34, 35). Wang et al. (36) reported that the surface roughness obtained by sandblasting titanium surfaces with alumina particles increased the adhesive strength between the titanium and the porcelain. Likewise, Lee et al. (37) observed that the highest surface roughness and adhesive strength were obtained in the sandblasted samples in their research on titanium substructures.

Some studies that examined titanium surfaces after sandblasting found alumina particles (35, 38, 39). Alumina particles, which loosely adhere to the surface after sandblasting, reportedly have a positive effect on adhesion without impacting the composition formed on the surface with ultrasonic cleaners (40). Wang et al. (35) stated that cleaning the titanium surface with steam removes oil and other similar residues that may form on the surface after sandblasting or occur with contamination. Thus, the adhesion strength of the samples prepared in this way is considerably increased.

In our study, the titanium disks were roughened using a Yb:Fiber laser in addition to the sandblasting and cojet process. The samples in the first group were scanned in the horizontal direction, the samples in the second group were scanned in a horizontal direction and subjected to two-sided surface roughening, and the samples in the third group were scanned in both the horizontal and vertical directions and according to the hypotenuse. Surface roughening was performed with triple scanning. According to the shear test data, the three-way scan group obtained the best bonding values. No statistically significant differences were found within or between the unidirectional scanning group, the bidirectional scanning group, and the three-way scanning group. As the number of scans with the fiber laser increased, the bond strength increased. The three-way scanning group demonstrated the maximum bond strength.

In our study, the bond strengths of the laser-roughened titanium disks with porcelain increased. Although the bond strength increased between the laser groups as the number of scans increased, there was no statistically significant difference between the bond strengths of the laser groups and the cojet, sandblasting, and control groups. After the shear test, the three-way scanning group showed the best average bond strength.

In general, surface wettability is thought to positively affect the adhesion between the metal surface and the porcelain. Li et al. (41) examined the surface contact angle and determined that the titanium control group samples were hydrophobic. In contrast, the titanium sample surfaces subjected to the micro-arc oxidation (MAO) technique were extremely hydrophilic. Yerokhin et al. (42) reported that by coating the metal surface using the MAO technique, a hydrophilic, rigid, and thick surface layer that adheres well to the metal surface can be formed.

According to the contact angle analysis in this study, the unidirectional group had the highest angle value (141°), whereas the control group had the lowest angle value (96°). Wettability is inversely proportional to the contact angle. Hence, this study compared the surface wettability between groups and determined that the unidirectional group was the most hydrophobic group. In contrast, the control group was the most hydrophilic group.

In the surface examination of the roughened titanium disc samples, which were made with the profilometer test, the highest surface roughness result was observed with a value of 2.74 μm in the unidirectional scanning group sample with the fiber laser. The lowest surface roughness result was seen in the control group sample with a value of 0.404 μm. In general, the surface roughness results were higher in the samples whose surface was roughened with fiber laser compared to all other groups. High surface roughness is desirable to some extent. As a matter of fact, although the highest surface roughness result was observed in single scanning samples with fiber laser, the most hydrophobic samples were also seen in single scanning samples with a fiber laser. In other words, too much surface roughness is not a desired situation. Although the lowest surface roughness results were seen in the control group samples, the most hydrophilic samples were also seen in the control group samples.

It was understood that the wettability alone was not sufficient for good bond strength, since the control group samples had the lowest bonding strengths, even though they were the highest hydrophilic samples. Here is the striking finding; Although the control group samples are hydrophilic, due to their low surface roughness, they also cause low bond strengths. In other words, very low surface roughness is also not desirable. In the samples of the sandblasting group, where the highest bond strengths were observed, average surface roughness results were observed compared to the other groups and were thought to have sufficient wettability.

Thus, it was thought that surface roughness results between 1.2 μm and 2.5 μm values and sufficient wettability gave better results for bond strengths. It was thought that excessive or excessively low surface roughness parameter results caused negative results in connection resistance.

In the analysis of the rupture surface of the samples in all groups after the shear bond strength test; While adhesive separation was not observed, cohesive separation was observed in 5 samples (2 control group samples, 2 cojet group samples, and 1 bidirectional fiber laser scanning group sample) and combined separation was observed in all other samples. Proportionally, 91.6% combined separation and 8.3% cohesive separation were observed after the rupture in all titanium-porcelain samples.

Conclusions

The conclusions and suggestions made within the scope of the results obtained in our study are as follows;

Within the results obtained in this thesis study, all of the mean bond values obtained from the other groups, except the control group (24.930 MPa), were higher than 25 MPa, which is shown as the minimum acceptable value for the bond strength between metal porcelain in the ISO 9693-1:2012 standard.

The roughening of the titanium infrastructure with the fiber laser technique positively affected the titanium porcelain bond strength and increased the average joint strength values.

The highest values of average titanium porcelain bond strength were obtained with fiber laser, triple scanning, and sandblasting roughening techniques. The highest average titanium porcelain bond strength value of 37.035 MPa was obtained in the sandblasting group samples.

As the number of scans increased in fiber laser etching groups, the binding values increased in parallel. It was observed that surface treatment with fiber laser gave positive results in terms of the strength of the titanium porcelain connection and increased the strength of the connection.

It was observed that the scanning group with the three-way fiber laser gave more successful results in terms of titanium porcelain connection compared to the other laser groups.

Fiber laser application was found to have a positive effect on the bonding for titanium disc specimens, but no statistically significant difference was found when compared with other groups.

In the SEM analysis examinations, fluctuations were observed on the surface of the titanium disc in fiber laser applications, increasing with the number of scans. Light micro cracks were observed in cojet applications, and craters and sharp crevices were observed in sandblasting applications.

Considering the results of the study, if one of the sandblasting methods with Cojet or Al_2O_3 is to be preferred for roughening the titanium infrastructure surfaces, although there is no statistical difference, Al_2O_3, which is a more traditional method, is recommended since the average bond strength is higher.

For roughening the titanium surface; Considering the difficulty of accessibility and cost calculation of fiber laser application, and because there is no statistically significant difference and the average bond strength obtained is similar, sandblasting and roughening can be preferred in clinical applications.

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