

Different Surface Treatments of Substructures on Bond Strength of Veneering Composite Resins

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Abstract: The aim of the study is to evaluate the effect of different surface treatments on the micro-Tensile Bond Strength (μ TBS) of hybrid composite to Lava Ultimate and Vita Ena-mic resin-nanoceramic CAD/CAM blocks/in this study for each CAD/CAM block 4 samples were prepared and divided according to surface treatment to 4 groups as follows: control, 50 μ m (Al_2O_3) airborne-particle, femtosecond laser and combination of 50 μ m (Al_2O_3) airborne-particle and femtosecond laser. One additional sample for each CAD/CAM block were prepared for each surface treatment group for SEM analysis. Filtek Z250 hybrid composite was adhered onto each sub group after application of universal single bond adhesive. Mikro Tensile Bond Strength (μ TBS) test for each sample was evaluated after 1000 thermal aging cycle. Bond strengths were analyzed through two-way ANOVA test. Tukey LSD test was used for multiple comparisons of the groups. For all groups, the control group showed the lowest bond strength values and the difference was statistically different ($p < 0.05$). Combination of sandblasting and laser groups showed highest bond strength values ($p < 0.05$). There is no statistical difference between sandblasting and laser surface treatment ($p > 0.05$). The SEM images were supported the (μ TBS) data. As a result of this study using these methods of surface treatments is expected to increase clinical success of these restorations.

Key words: Bond strength, CAD/CAM, surface treatment, femtosecond laser, SEM analysis, control group

INTRODUCTION

The pleasing esthetics of all-ceramic restorations explains their increasing popularity in cosmetic dentistry. In addition, the biological properties of currently available all-ceramic systems make them suitable for tooth reconstructions and high-quality esthetic restorations for many clinical indications. Furthermore, owing to improvements in their mechanical properties, all-ceramics can now also be used to restore both single and multiple tooth defects (Shillingburg *et al.*, 1997). By reducing laboratory procedures CAD/CAM systems provided faster solutions. Variety of materials used for CAD/CAM make possible to obtain substructures from different materials or to prepare the full restoration from one block. Using composite resin as a veneer material is a new and alternative method (Shillingburg *et al.*, 1997; McLean, 2001). A number of surface pretreatments have been developed that mechanically facilitate the bonding between resin and ceramic surfaces. The recommended method for conditioning the surfaces of ceramic restorations is to treat them with hydrofluoric acid and

subsequently apply a silane coupling agent to ensure strong bonding (Sjogren *et al.*, 1999; Holand *et al.*, 2000). Hydrofluoric acid creates a retentive surface for micro mechanical bonding via the preferential dissolution of the glassy phase and the applied silane-coupling agent increases the wettability of the surface and promotes the formation of covalent bonds between the methacrylate groups of the resin and the silica in the ceramic (Kern and Thompson, 1995; Zel *et al.*, 2001).

Airborne-particle abrasion is a conventional surface treatment method that creates a rough, irregular surface and improves micro mechanical retention by increasing the surface area and the adhesion energy of resin to all-ceramics. Abrasion performed with aluminum oxide particles under pressure decreases the surface tension and improves the wettability of silane-coupling agents on ceramic surfaces (Ozcan and Vallittu, 2003).

Etching the surface of zirconia with Nd: YAG and Erbium-doped YAG (Er:YAG) lasers strengthened the subsequent bonding more than sand blasting did. Neodymium-Doped Yttrium Aluminum Garnet (Nd:YAG) lasers have been used to roughen the surfaces of solid

ceramics to attach veneering porcelain or resin cements, for tooth bleaching, to reduce tooth sensitivity and to remove caries. Lasers have been used in dental clinical practices, since, the 1960's but a number of new applications have recently come to the fore (Usumez and Aykent, 2003; Meerbeek *et al.*, 2003).

Ultrashort-Pulse Laser Systems (UPLS's) works through cutting with minimal collateral damage, allowing the ablation of thin layers with extreme precision and high reproducibility with no thermal side effects and used in different fields including medicine, biology and the micromachining of materials. UPLS's it has been reported in previous studies that femtosecond laser treatment improves the Shear Bond Strength (SBS) of brackets attached to enamel surfaces. This kind of treatment should therefore, also be suitable for all-ceramics (Meerbeek *et al.*, 2003; Dausinger *et al.*, 2004).

Although, the advantages and disadvantages of different types of surface treatments applied to different ceramics have been reported by many studies, the ideal treatment to strengthen bonding between resin and ceramic surfaces is under investigation. The present study was therefore, conducted to evaluate the effects of various surface treatments on the surface structure of resin-nano ceramic CAD/CAM block and on their bonding to hybrid composite via. Scanning Electron Microscopy (SEM). The null hypothesis was that the different surface treatments would not affect the bond strength of the composite resin and resin-nano ceramic CAD/CAM block.

MATERIALS AND METHODS

A total of Usumez and Aykent (2003) square shaped Resin-Nano Ceramic CAD/CAM samples with $10 \times 12 \times 4$ mm in diameter was achieved from each resin nano ceramic CAD/CAM blocks material lava ultimate (3 m ESPE, Neuss/Germany) and vita enamic (Ivoclar-VivaDent, schaan/Liechtenstein-Germany) by the use of diamond saw (Isomet, Buehler, Lake bluff, il, USA) at 150 rpm in this study.

According to surface treatment process resin Nano Ceramic CAD/CAM sample were divided into Holand *et al.* (2000) subgroups control group (C) in which no additional surface treatment was performed, Sand Blasting group (SB) by which samples were sandplasted with $50 \mu\text{m}$ under 2 bar pressure Al_2O_3 particles (Korox, BEKO, Bremen, Germany) by using the sand blasting machine Renfert Basic Master, Buck-Inghamshire, UK), Femtosecond Laser Group (FL) in which a pulse rate of 2 kHz was used by laser marking device (q Mark-Quantronix, NY, USA). Samples were

treated with femto second by a system based on a titanium: sapphire oscillator which produces 90 fs, 750 mW pulses at a wavelength of 810 nm for 60 sec at 45° angulation and the 4th group is (combination group) which is a combination of sandblasting and laser surface roughening. surface roughening was applied first by sand-blasting process and then laser application was done by using the parameters determined in the group of sandblasting and laser. ($n = 1$) resin nano ceramic CAD/CAM sample were used for each surface treatment group and ($n = 1$) resin nano ceramic CAD/CAM sample for (SEM) analysis.

Before surface treatment process, carbide sandpapers paper (600-1000 grit) and a polishing machine (Minitch 233; Presi, Grenoble, France) used with water cooling for polishing. The bonding surfaces and cleaned ultrasonically in distilled water for 10 min to remove any surface contaminants (Biosonic UC 50; Coltene Whaledent Inc., Cuyahoga Falls, OH).

For bonding of composite resin to CAD/CAM block samples a thin layer of single bond universal adhesive (Universal Single Bond, 3 m ESPE, Neuss, Germany) was applied with a brush on the surface of each surface-treated specimen. The specimens were then dried in air using oil-free compressed air. To apply the resin, the treated ceramic specimens were placed in a plastic molds. The filtek Z 250 hybrid composite (3 m ESPE, Neuss, Germany) applied to CAD/CAM samples in 1 mm layers with a total height of 4 mm and polymerization was done by a light-emitting diode unit (built-in C, Woodpecker, Guangxi, P.). According to the recommendation of the manufacturer polymerization was done for 40 sec with a light intensity of 600 mW/cm^2 in every aspect in contact with the samples. With a flat glass a small pressure applied to the upper surface of the composite. In the final layer and after a pre-polymerization of 40 sec with a 40 sec light curing all over the resin-CAD/CAM block samples the polymerization process was completed.

Before the samples thermocycled (Thermal Cycler Tester; Dental Teknik, Konya, Turkey) for 1000 cycles between 5 and 55°C , the dwell and transfer times were 30 and 10 sec transferring time between the two pools the bonded specimens were stored in distilled water at 37°C for 24 h and subsequently. The micro-tensile test were done by micro-tensile test device (the Micro Tensile Tester, Bisco, Schaumburg, il, USA) after preparation of a total of 1×1 mm 20 micro bars for each test group ($n = 20$) by using 150 rpm diamond saw device (Isomet, Buehler, Lake bluff, il, USA). Bond strengths were measured by application of 1 mm tensile force per minute until a separation was occurred and conversion of values recorded into MPa were done (Table 1 and 2).

Table 1: Means of Microtensile values of different surface treatment groups (MPa)

Samples	Sandplast+			
	Laser	Laser	Sandplast	Control
Vita enamic (n = 20)	29.87±5.43	33.99±4.57	32.15±4.62	19.35±4.56
Lava ultimate (n = 20)	24.82±3.79	33.39±3.79	27.61±4.88	16.65±3.85

Table 2: Statistical Identification of the amount of microtensile bond (MPa)

Surface treatment	n	Ortalama	SD Sapma
Laser	40	27.34	5.282
Sandblast+Laser	40	33.69	3.88
Control	40	18.00	4.38
Sandblast	40	29.88	5.22
Total	160	27.22	7.46

For the SEM analysis, the ceramic specimens were first sputter coated with gold-palladium particles and Three different magnifications (1000, 2500 and X5000) used for studying the selected test samples by SEM device (Scanning Electronic Microscope, Jeol Ltd., JSM-5600, Tokyo, Japonya).

Two-way ANOVA was used to evaluate the effects of the ceramic type and the surface treatments on the microtensile bond strength. Tukey LSD multiple comparyson test was used for subgroups comparison. Kolmogorov Simirnov test used to test the suitability of the data for normal distribution. The $p < 0.05$ was considered significant.

RESULTS AND DISCUSSION

Combination of sandblasting and laser groups showed highest bond strength values obtained ($p < 0.05$). Lowest bond strength values obtained in the control group and the difference was statistically different ($p < 0.05$). There is no statistical difference between sandblasting and laser surface treatment ($p > 0.05$). The two-way ANOVA tests revealed that the microtensile bond strength was affected both by the surface treatment and the type of ceramic used ($p < 0.05$). Surface treatment and the block interaction difference was not significant ($p > 0.05$). The bond strength strength values obtained in both CAD/CAM block samples in the control group was the lowest. The highest values of bond strength was found in the groups surface treated with combination of $50 \mu \text{ Al}_2\text{O}_3$ and femtosccond lazer for two CAD/CAM block samples. Sandblasting and laser groups followed these groups. The bonding strength values of vita enamic laser group and lava ultimate sandblasting group was statistically similar (Fig. 1 and 2).

SEM images observation of the surface treated of groups reveals that control groups images are fairly flat. SEM images of other groups were observed to have a

surface structure completely different from the surfaces of the control group. Surface structure of sandblasting and laser combination group observed to be more porous and cracked.

Null hypothesis (no effect) is rejected because bonding of composite resin to CAD/CAM block samples affected by the surface treatment process. Connection between cement and dental tissue and restorative material determines the clinical success of complete ceramic restorations prepared as inlays, onlays or laminate veneers with feldspathic content (Leinfelder *et al.*, 1989; Kelly *et al.*, 1996). Ceramic surface treatment Mechanically by sandblasting with aluminium oxidewith different particle size or etching with acidare recommended for increasing the wetness of ceramics and lead to a better resin bond strength through changing the surface structure which provides micro-mechanical retension (Leinfelder *et al.*, 1989; Kelly *et al.*, 1996; Anusavice *et al.*, 2007).

In this study, $50 \mu \text{ Al}_2\text{O}_3$ was preferred for sandblasting of hybrid ceramic CAD/CAM surfaces due to it's usefor roughening ceramic surfaces in previous studies which lead to increasing the bonding strength of the resin composite by increasing surface roughness in ceramic materials which lead to increasing of surface energy and thus increasing wetness of ceramic (Kem and Wegner, 1998; Akyil *et al.*, 2010).

Direct clinical use of ultrashort-pulse laser systems are not suitable. However, many studies have reported on the bond strength of dental tissues subjected to treatments using a UPLS. Also, reported in studies stronger bonding of brackets to enamel treated with a UPLS rather than other laser systems. The ablation from UPLS's is induced by a plasma which imparts less thermal damage than do the longer pulses of ER: YAG lasers. The bonding of resin cement to porcelain treated with a UPLS not enough been studied. A large number of parameters determine the suitability of modern UPLS's (with scanning systems) for dental applications. Nevertheless, the ablation rates afforded by these systems make them promising for dental applications. The UPLS significantly altered the irradiated area and lead to increase in bond strength in the study evaluated the SBS of repairs in porcelain conditioned with a UPLS (760 and 900 mW) for 10 sec or with 10% HF acid for 2 min (Wolfart *et al.*, 2007; Baehr *et al.*, 2013).

SEM images of the FS surface treated group revealed large surface fissures which were more homogenous and more uniform. Previous studies reported that increasing surface roughness in ceramic materials increases surface

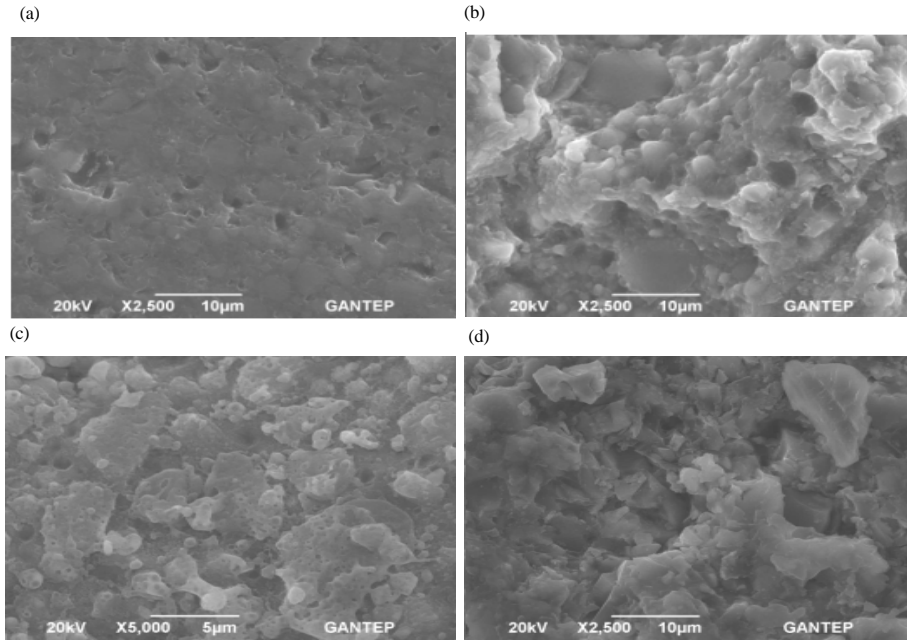


Fig. 1: a-d) SEM image of Lava ultimate showing surface tomography of the control, sanblast, laser and combination of laser and sandblast, respectively at X2500 magnification

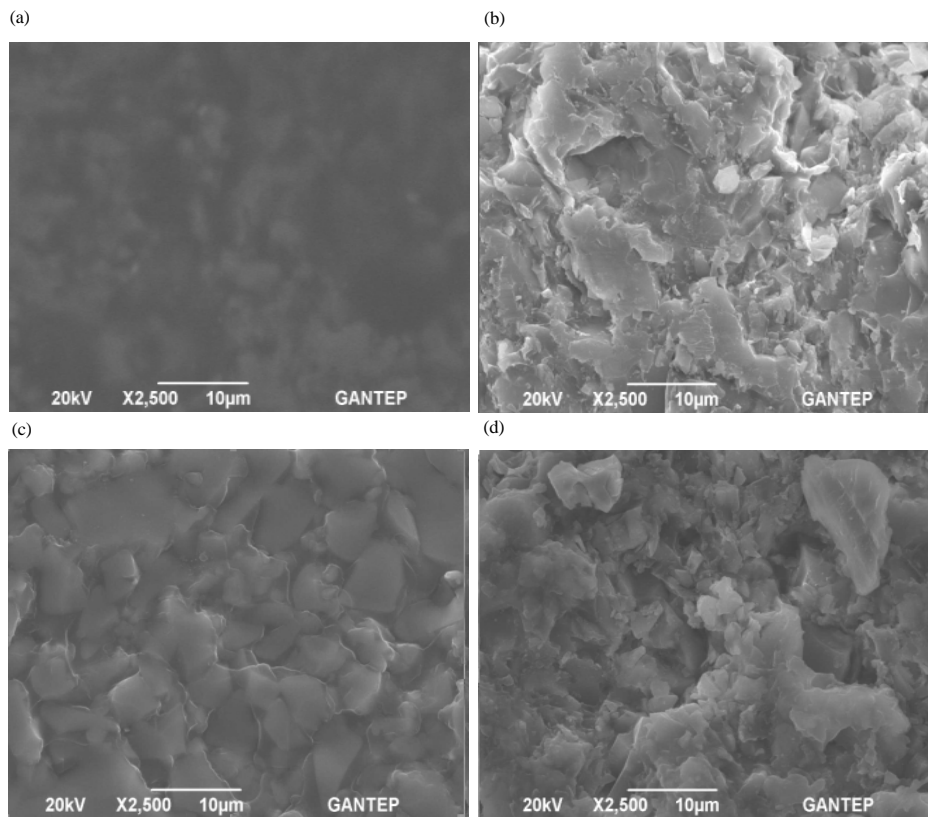


Fig. 2: a-d) SEM image of Vita enamic showing surface tomography of the control, sanblast, laser and combination of laser and sandblast, respectively at X2500 magnification

energy and thus increases the bonding strength of the resin composite by increasing wetness of ceramic. In our study, there was an increase in bonding strength in groups treated with femtosecond laser but the highest bonding value was found when the laser used after sandblasting with 50 μ Al_2O_3 and SEM images support these results (Baran *et al.*, 2001; Bindl and Mormann, 2005).

The surface characteristics of porcelain have a major influence on the quality of the contact between the adhesive and the solid ceramic surface. The adhesive wets and spreads over the surface, penetrating into the pits formed by roughening or chemicaletching. Hydrofluoric acid treatment removes the glass matrix and the second crystalline phase while silane-coupling agents increase the wettability of the surface, forming structural layers that promote bonding. The type of porcelain used affects the composition and physical properties of the ceramic and determines the type of adhesion layer formed on the surface. Physical and chemical changes can increase bond strength by affecting surface energy and wetting ability of the surface as reported in previous studies (Bindl and Mormann, 2005; Denry, 1996; O'Brien, 2002).

Chemical bonds formed between silane containing primer in single bond universal adhesive and vita enamic and lava ultimate hybrid ceramics had been reported by previous studies, chemical links formed between the methoxy groups of the silane containing primer and the SiO_2 and polymer network components of vita enamic and lava ultimate CAD/CAM hybrid ceramics. Chemical links also formed between methoxy groups of silane molecules and methacrylate groups and SiO_2 of resin composites on the other hand and bonding strength increase had been occurred due to these chemical links. It was concluded that the difference in composition made the bond strength values in vita enamic to be higher than Lava Ultimate due to more resin content in vita enamic CAD/CAM hybrid ceramic (O'Brien, 2002; Rinke and Huls, 1996).

CONCLUSION

Clinical success of the restorations increased due to using these methods of surface treatments. Increasing the bond strength of composite resin to ceramic surfaces using UPLS seems to be a suitable conditioning method. Further investigations for various parameters are required.

LIMITATIONS

Clinical success of full ceramic restorations adhered to resin with the help of silane may be influenced in the

long term because it does't take to account preparation design and the three-dimensional geometry clinical trials of the restoration which are usually not considered all in vitro studies.

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