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## Effect of the Er, Cr: YSGG Laser Parameters on Shear Bond Strength and Microstructure on Human Dentin Surface

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### 1. Introduction

The developments of laser technology have enabled their use in multiple dental procedures, such as soft tissue operations, composite restorations, tooth bleaching, root canal irrigation, caries removal and tooth preparations with minimal pain and discomfort (Turkmen et al., 2010). Recently, the Er,Cr:YSGG laser was recommended for minimally invasive purposes due to its precise ablation of the enamel and dentin without side-effects to the pulp and surrounding tissues. It has a 2780-nm wavelength and absorbed strongly by both water and hydroxyapatite. The sudden evaporation of bound water causes micro-explosions that blast away tiny particles of the tooth (Obeidi et al., 2010).

Previous studies reported that irregularities and the crater-shaped appearance of ablated dentin was comparable to the dentine surface after acid etching, which might promote micromechanical interlocking between dental restorative materials and the tooth surface (Visuri et al., 1996; Armengol et al., 1999; Martínez-Insua et al., 2000; Carrieri et al., 2007; Gurgan et al., 2008). Despite its efficiency, reports on the bond strengths of composite resin to a tooth substrate prepared by a laser are often confusing and contradictory. Some studies reported higher bond strengths to laser-prepared dentin than to acid-etched dentin (Visuri et al., 1996; Carrieri et al., 2007). Others have reported significantly lower bond strengths on laser preparation (Armengol et al., 1999; Martínez-Insua da Silva Dominguez et al. 2000; Gurgan Kiremitci et al. 2008) and others have reported no significant differences (Abdalla & Davidson, 1998).

Generally there are three adhesive systems. The first uses 30–40% phosphoric acid to remove the smear layer (etch-and-rinse (ER) technique). This bonding mechanism to dentin

depends on the hybridization of the resin within the exposed collagen mesh as well as to the dentin tubules (Abdalla & Davidson, 1998), creating a micromechanical interlocking of the resin within the exposed collagen fibril scaffold. The second is the “self-etch” adhesives (SEA) which employs acidic monomers that simultaneously condition and prime dentin. The smear layer remains partially but is used to hybridize with the underlying dentin (Perdigao et al., 2000). The last is an all-in-one system, but the stability of the bond strength decreases with time because it contains many hydrophilic monomers.

According to previous reports, a tiny flake surface formed by the laser irradiation of dentin can be removed with 30-40 % acid etching, but the primer of SEA cannot. This study hypothesized that the primer of SEA cannot improve the free surface energy of irradiated dentin. Therefore, it is unable to make a proper environment for sufficient bond strength. The null hypothesis was that the shear bond strength with “self-etch” adhesives (SEA) did not show a difference from that with the etch-and-rinse (ER) technique after a pretreatment with a laser in dentin.

This study examined the shear bond strength of a hybrid composite resin bonded with two different adhesive systems to the dentin surfaces prepared with Er,Cr:YSGG laser etching, and evaluated the morphologic structure of de-bonded dentin surface after Shear Bond Strength (SBS) Test by scanning electron microscopy.

## 2. Materials and methods

### 2.1 Tooth preparation

Tables 1 and 2 list the materials used in this study and the study design, respectively. Twenty four freshly extracted caries and restoration-free permanent human molars stored in distilled water were used. The teeth were embedded in improved stone with the occlusal surface of the crown exposed and parallel to the base of the stone, and the embedded teeth were sectioned at one third of the occlusal surfaces to expose the dentin surface. Each tooth was wet-ground with 320-grit silicon carbide paper and polished with 1200-grit to obtain a flat dentin surface. The specimens were stored in distilled water at 37 °C. The teeth were divided randomly into two groups, control and laser irradiated groups. The control groups without laser irradiation were divided randomly into two subgroups (SE bond and Single bond), and the laser irradiated groups were divided into four subgroups (SE bond with 1.4 W, 2.25 W and Single bond with 1.4W, 2.25W).

### 2.2 Laser irradiation

The Er,Cr:YSGG laser (Waterlase, BioLase Technology, Inc., San Clemente, CA) with a 2780 nm wavelength and 20 Hz of power with a sapphire tip was used. Laser irradiation was performed on the dentin surface with either 1.4 W or 2.25 W. The flattened dentin surfaces of the teeth were irradiated at 90° in non-contact mode with a fixed distance of 6 mm away from the laser tip in a sweeping motion to achieve an even surface coverage by overlapping the laser impact. The laser handpiece was attached to a modified surveyor to ensure a consistent energy density, spot size, distance and handpiece angle.

### 2.3 Bonding procedures

In all groups, including laser irradiated groups and control groups, the bonding procedures recommended by the manufacturer' instruction were followed strictly. In the single bond

adhesive system, etching procedures were conducted using 37 % phosphoric acid (3M ESPE, St. Paul, MN, USA) for 15 seconds followed by rinsing with a water spray for 15 seconds, and blot dried, and bonded and light curing for 15 seconds. In the Clearfil SE bond system, a primer was applied to the dentin surface for 10 seconds followed by bonding and light curing, as shown table 1.

After light curing the bonding resin, a Teflon tube (GI tech, Seoul, Korea) with an inner diameter of 2 mm and a height of 2 mm was attached to each dentin surface and filled with A3 Body Shade of the hybrid composite Resin (Filtek Supreme Plus 3 M ESPE, MN, USA) followed by light curing for 40 s. After light curing, the teeth were stored in water at 37°C for 24h before the Shear Bond Strength (SBS) Test.

Brand name	Manufacturer	Material type	Composition	Application procedure
Clearfil SE bond	Kuraray, Tokyo, Japan	Self etching adhesive system	Primer: MDP, HEMA, N,N-Diethanol p-toluidine, water Bonding resin: Bis-GMA, CQ, HEMA, MDP, Micro filler	Apply primer and leave 20 seconds, air blow, apply bonding system
Single bond	3M ESPE, St. Paul MN, USA	Etch and rinse adhesive system	Bis-GMA, HEMA, Water, UDMA, Ethanol, Polyalkenoic acid copolymer	Acid-etch for 15 seconds, rinse, apply adhesive, gentle air blow, light curing for 10 seconds.

Table 1. Materials used in this study.

Laser intensity	Adhesive system	
	Single bond	SE bond
Control (no laser)	Group A	Group D
1.4W	Group B	Group E
2.25W	Group C	Group F

Table 2. Classification of the experimental groups.

#### 2.4 Shear Bond Strength (SBS) test

Before loading, the tube mold was removed carefully with a sharp blade and the specimens were then placed in a custom-made fixture mounted on a Universal Testing Machine (INSTRON Model 5562, Norwood, MA). The specimens were loaded to failure under

compression using a knife-edge loading head at a cross-head speed of 1 mm/min. The investigator, who was blinded to the group treatment, performed the load testing procedure. The maximum load to failure was recorded for each sample and the Shear bond strength (SBS) was expressed in megapascals (MPa), which was derived by dividing the imposed force (Newtons) by the bonded area (mm<sup>2</sup>).

## 2.5 SEM evaluation

To evaluate the surface (treated with Etching, Laser, and Laser + Etching) before bonding and the fractured surface between the composite resin and dentin side after measuring shear bond strength, the representative dentin specimens were selected randomly and mounted on aluminum stubs. The samples were then sputter-coated with gold palladium and examined by scanning electron microscopy (SEM) (ISI SX-30 Cambridge, MA, USA) with 1000X magnification.

## 2.6 Statistical analysis

The obtained shear bond strength data were analyzed by two way ANOVA with a level of significance of  $P < 0.05$ .

## 3. Results

### 3.1 Shear Bond Strength (SBS) evaluation

The Mean SBS $\pm$ SD (MPa) of each group followed by a Single bond was 7.35 $\pm$ 1.65, 7.61 $\pm$ 1.50 and 6.34 $\pm$ 1.39, respectively, and the Mean SBS $\pm$ SD (MPa) for each group followed by a SE bond was 7.73 $\pm$ 1.17, 7.88 $\pm$ 2.77, and 6.48 $\pm$ 1.77, respectively (Table 3), but there were no significant differences between the groups. The maximum and minimum SBS was observed in groups E and C, respectively. The maximum and minimum SBS in the Single Bond group was observed in groups B and C, respectively. The maximum and minimum SBS in the SE Bond group was observed in group E and F, respectively (Figure 1). Some samples dislodged and de-bonded during preparation for the shear bond test due to the uneven samples distribution.

Adhesive system	Single bond			SE bond		
	Control	1.4 W	2.25 W	Control	1.4 W	2.25 W
Sample	Group A	Group B	Group C	Group D	Group E	Group F
Mean (MPa)	7.35	7.61	6.34	7.73	7.88	6.48
SD	1.65	1.50	1.39	1.17	2.77	1.77

Table 3. Comparison of the mean shear bond strength of the control and laser irradiated groups (Single bond and SE bond). Mean value (MPa) and standard deviation (SD) of the Shear Bond Strength (SBS) Test for the specimens treated with the Er,Cr:YSGG laser are shown but there were no significant differences between the groups ( $P > 0.05$ ).

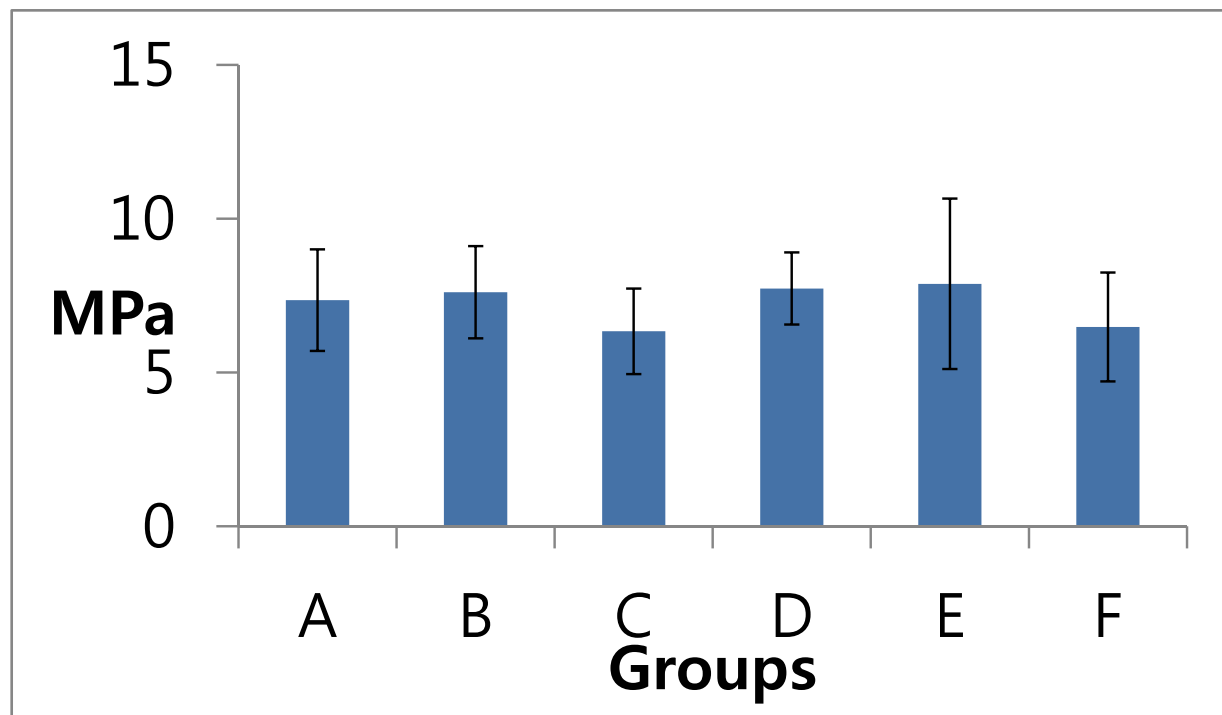


Fig. 1. Shear bond strength between the experimental groups.

### 3.2 SEM (Scanning Electronic Microscopy)

SEM revealed the fractured surface between the composite resin and dentin side, as shown in Figure 2. The representative SEM evaluation showed that thick layers of the adhesives were found on the fractured de-bonded surface of the composite resin (Figure 2-B, D, F). On the other hand, the fractured bonded surface of the dentin by the Clearfil SE bond adhesive treatment, as a result of the shear bond strength test followed by 37% phosphoric acid etching of some of the residues of the adhesives remaining on the surface of dentin side after the shear bond test (Figure 2-A, C, E).

Regarding the failure mode of the control group in the types of adhesive system, some residual chips were found (A) on the surface of dentin side fractured after 37% phosphoric acid etching and single bond adhesion but the fractured bonded surface of dentin by the Clearfil SE bond adhesive treatment as a result of the shear bond strength test followed by 37% phosphoric acid etching showed thick layers of composite resin attached to the dentin after the failure test (D).

In the irradiation groups, 1.4 W of irradiation followed by the Single bond procedure left the fractured dentin surface with small particles after the failure test (B). On the other hand, the fractured dentin surface irradiated with a power intensity of 2.25 W had exposed dentinal tubules after the test (C). On the other hand, 1.4 W of power intensity irradiation followed by the SE bond procedure revealed exposed dentinal tubules after the failure test (E), whereas 2.25 W of power intensity irradiation followed by the SE bond procedure resulted in a thick layer of composite resin fractured cohesively on the dentin side of the fractured surface (F).

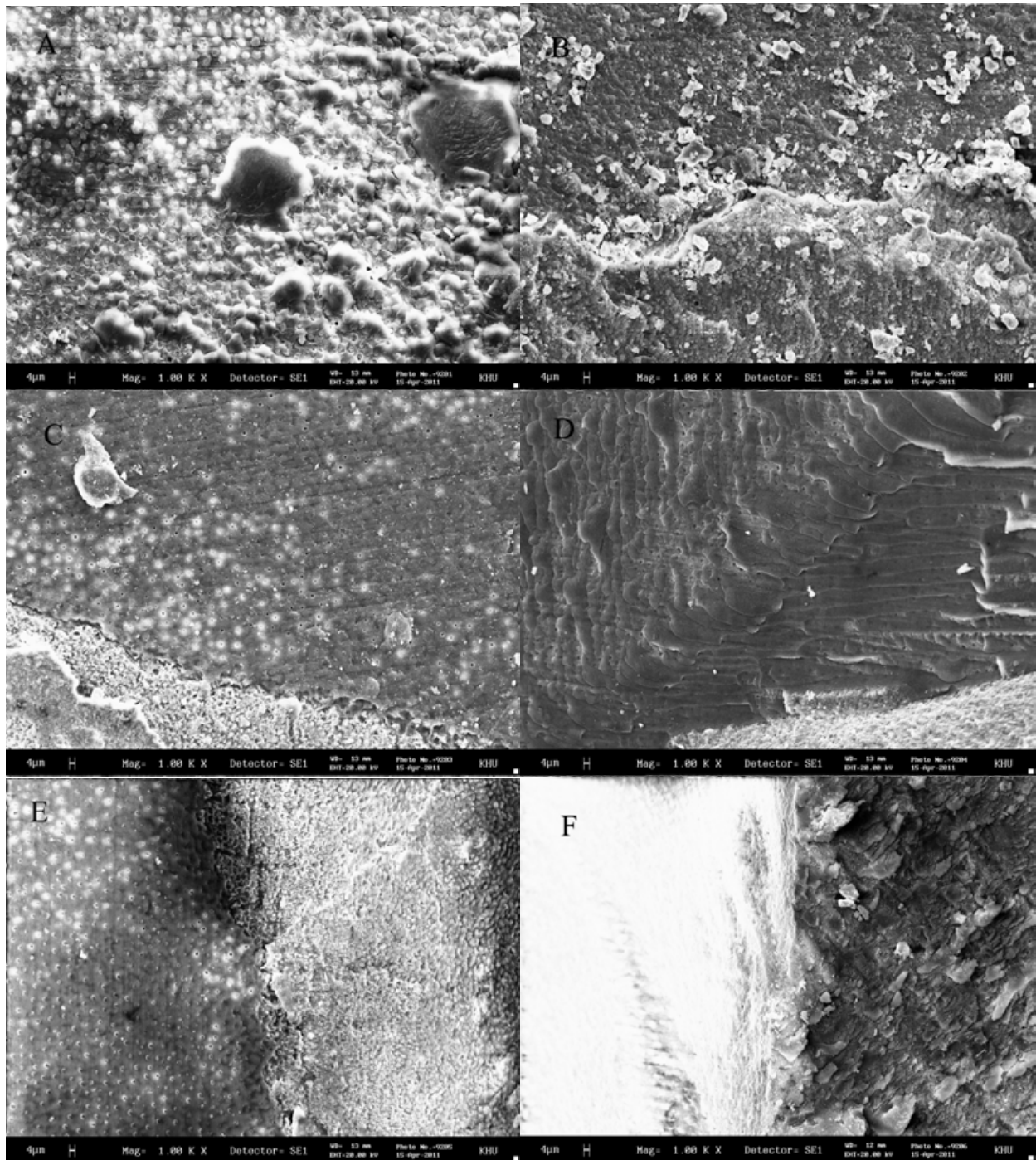


Fig. 2. SEM images (X1000) showing the fractured surface between the composite resin and dentin side. The fractured bonded surface of dentin by the single bond adhesive treatment as a result of the shear bond strength test followed by 37% phosphoric acid etching (A), and 1.4 W of power intensity irradiation followed by a Single bond procedure (B), and 2.25 W of power intensity irradiation followed by a Single bond procedure (C) were evaluated by SEM. The fractured bonded surface of the dentin by Clearfil SE bond adhesive treatment as a result of the shear bond strength test followed by 37% phosphoric acid etching (D), and 1.4 W of power intensity irradiation followed by the SE bond procedure (E), and 2.25 W of power intensity irradiation followed by the SE bond procedure (F) were evaluated by SEM.

#### 4. Discussion

Recently, Er:YAG and Cr:YSGG lasers were introduced in dentistry. For physical and medical reasons, they are used for the treatment of hard tissue. The advantage of an Erbium wave is that it is well absorbed by water and dental hard tissue. Although dentin contains a high proportion of water, the depth is shallow for laser transmission. The strong absorption of water reduces the level of heat during tooth preparation. As water absorbs laser radiation better than dental hard tissue, it reduces the increasing temperature of the tissue during the preparation. Water reaches the boiling point and causes microexposure of the tooth. This action breaks up the surrounding tissue into small pieces and dissipates them at the same time. As this explosion occurs in water, it is so-called a preparation induced by water. Although most radiation is absorbed in water, a certain amount of heat transmission is unavoidable. Therefore, a water spray is used for cooling. The proper amount of water spray prevents pulpal damage. As heat transmission normally depends on the pulse shape and pulse maintenance time, the amount of water varies according to the laser system and treatment itself. A lack of enough water spray causes pulpal damage.

In contrast to initial studies, there was no immunological difference between the laser system and conventional etching method. Moreover, the positive effect of the laser to regenerate pulp was discussed. Essentially, cavity preparation should be performed with the proper instruments with the "method of minimal pulse energy" according to the manufacturer's protocol. This is because the preparation should be performed with the capability to minimize additional damage.

The bond strength of the adhesive system is one of the major factors for the success of restorations. Therefore, this restoration parameter can be measured accurately by the bonding test. The bonding of resin composite material to the tooth structure prepared with different types of lasers has been reported (Visuri et al., 1996; Armengol et al., 1999; Martínez-Insua et al., 2000; Carrieri et al., 2007; Ekworapoj et al., 2007; Lee et al., 2007; de Carvalho et al., 2008; Gurgan et al., 2008; Chou et al., 2009). These studies reported variable results comparing the bond strengths of composite resin to a laser-prepared and acid etched dentin surface, and suggested that the laser preparation is more effective than etching in bonding strength of the composite resin to the tooth structure (Obeidi et al., 2010; Turkmen et al., 2010; Navimipour et al., 2011). This study compared the *in vitro* SBS of two different adhesive systems to dentin treated with an Er,Cr:YSGG laser. These results showed that the type of adhesive system had no effect on SBS, whereas the laser intensity was a criterion to be considered.

Generally, the manufacturer of Er,Cr:YSGG lasers recommended 2.25 to 2.5 W for laser etching but other studies used 2 to 3 W referring from their results of a pilot study to obtain the proper effects (Çalışkan et al., 2010). The data from this study showed that the shear bond strength (SBS) of dentin treated with 1.4 W, was higher than that of SBS with 2.25 W, which was recommended by the manufacturer. These results were different from other study results showing that the shear bond strength of laser etching (1.25 W or 3 W) was higher than the diamond bur (Gurgan et al., 2008). The distance between the laser tip and laser irradiated dentin surface may not be strict in this kinds of study designs. On the other hand, in this experiment, the sweeping motion of the laser irradiation 6 mm away from the dentin surface was used before the application of adhesive procedures to employ consistent defocused irradiation with lower intensity. Therefore, the difference in the effect between



the distance and irradiation time of the laser could not be performed and compared with other studies in this study (Chou et al., 2009; Obeidi et al., 2009; Obeidi et al., 2010).

Based on previous studies using Er,Cr: YSGG laser irradiation on the tooth structure, normal range of power intensity (2.5 W) was utilized on the surface of dentin while high power intensity of the laser (higher than 3 W) was more effective on the bonding strength of the enamel surface (Obeidi et al., 2010), (Dunn et al., 2005). On the other hand, Tagami *et al.* reported that 70mJ (1.75 W) of low laser intensity irradiation on the dentin surface followed by SE bond showed a higher micro shear bond strength than with 150mJ (3 W) (de Carvalho et al., 2008), which is similar to the present experiments. According to the manufacturer's recommendation for laser etching on dentin surface, it was decided to follow the laser protocol mode using two power intensities (2.5 W and 1.25 W). If a power intensity of 2.25 W and 1.4 W are converted to the mJ scale, they could be approximately 112.5 mJ and 70 mJ, respectively. In the present study, 1.4 W of laser intensity irradiation showed the highest shear bond strength between the composite resin and bonded dentin surface with the two types of adhesive systems (Single bond and Clearfil SE bond). Consequently, the SE primer of the SE bond probably could not remove the superficial layers of the irradiated dentin completely. On the other hand, phosphoric acid of three steps adhesive or two steps adhesive could remove most of the superficial layers of the irradiated dentin. In addition, one study reported the advantages of mechanical instruments or combining acid etching and mechanical instruments in removing the superficial layers (Obeidi et al., 2009).

With the respect to the micromorphological changes after acid etching and laser etching, SEM revealed different characteristic features from those found in conventional acid etched surfaces. The dentin surfaces irradiated with the Er,Cr:YSGG laser had a scaly, irregular, and rugged appearance compared to the acid etched dentin surface (Chou et al., 2009). In addition, with the higher laser intensity or longer irradiation time, the condition was worse in terms of the irregularity of the dentin surface, which has a close relationship with the bonding strength of the composite resin to dentin.

The control groups showed a higher SBS than the 2.25 W of laser irradiated groups in all bonding systems, but, SEM evaluation revealed more adhesive chips remaining on the control group than on the 2.25 W laser irradiated group. On the other hand, thick, rough, and irregular collapsed composite resins were estimated on both the control and 2.25 W laser irradiated groups. SEM examinations of the two kinds of laser intensity irradiation (1.4 W and 2.25 W) revealed that the 1.4 W laser intensity group had more adhesive chips remaining on the dentin than the 2.25 W laser group in the Single bond adhesive system, which is opposite to that observed in the Clearfil SE bond system.

One possible explanation is that the thermomechanical effects of higher laser intensity probably have extended into the subsurface dentin and undermined the integrity of the resin-dentin interface resulting in a lower bond strength. The formation of fissures or cracks in the subsurface dentin might be a start point for the failure of resin-dentin adhesion. Obviously, all irradiated groups were affected by thermo-mechanical effect, but 1.4 W and 2.25W were all affected by thermomechanical effects. On the other hand, 1.4 W laser intensity might not be seriously affected. Laser irradiation with a high intensity may obstruct the dentinal tubules by melting and fail to produce a good hybrid layer. Whereas, laser irradiation with a low intensity may leave the dentinal tubule open and facilitate the infiltration of bonding agent. This may account for the lower SBS values with 2.25 W irradiated dentin than with 1.4 W laser intensity.

These results suggest that the acid etching of lased dentin can reinforce the hybrid layer and formation of resin tags, but the acidic monomer of SE bonds cannot function at its best due to the obstruction of dentinal tubules and denatured collagen fibrils network and the absence of smear layer. These results were not the same as expected. A self etching adhesive system and two steps adhesive system showed a similar SBS because the resin tags only contribute to the bond strength in small portions (15%) and the poorer quality of the hybrid layer appears to be the main reason for the lower bond strength.

## 5. Conclusion

An Er,Cr:YSGG laser was used to determine if the laser can increase the shear bond strength between the composite resin and surface treated dentine surfaces. On the other hand, the pretreatment of dentin with an Er,Cr:YSGG laser does not affect the shear bond strength of the two different adhesive systems under these experimental conditions. In addition, the shear bond strength of two different adhesive systems in dentin treated with a 1.4W laser intensity was higher than that treated with 2.25W but the difference was not significant.

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