



## Bond Strength of Glass Carbomer Cements on Laser-Etched Dentin Surface

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### Abstract

**Purpose:** The aim of this study was to evaluate the shear bond strength (SBS) of glass ionomer and glass carbomer cements on Er,Cr:YSGG laser or acid-etched human dentin surface.

**Materials and Methods:** One hundred and twenty extracted caries-free human third molar teeth were selected and occlusal enamel was removed until dentinal surface was exposed. The teeth were randomly divided into four main groups (n=30). Then four main groups were divided into three subgroups. For the dentin surface treatment; first group was etched with 37% phosphoric acid or 20% polyacrylic acid, second group was etched with Er,Cr:YSGG laser and third group was not etched served as the control group. Fuji IX GP, Fuji II LC, Dyract XP and GCP restorative materials were applied to dentin surface as 4x4 mm cylindrical block. After storing the specimens in artificial saliva at 37°C for 24 h, SBS of the restorative materials were measured with Shimadzu AGS-X 50 kN testing machine at crosshead speed of 0.5 mm/min. The fractured surfaces were examined in stereomicroscope and SEM. Statistical analysis was made using SPSS program for Windows 21.0 with Post Hoc LCD test.

**Results:** For Fuji IX, there is no significant difference between acid-control and laser-control groups ( $p>0.05$ ); the highest bond strength values were observed with etched condition ( $p<0.05$ ). For Fuji II, there is no significant difference between acid-control groups ( $p>0.05$ ); laser application showed the lowest bond strength values compared to other surface conditions ( $p<0.05$ ). For Dyract XP, acid and laser groups showed better bond strength values than control group ( $p<0.05$ ); no significant difference was found between acid-laser groups ( $p>0.05$ ). For GCP group, there was no significant difference between in all conditions ( $p>0.05$ ). Moreover, in all different conditions of the dentin surface treatment, bond strength values were observed highest in Dyract XP specimens and lowest in GCP specimens ( $p<0.05$ ).

**Conclusions:** Application of laser on dentin surface weren't found effective on bond strengths of the restorative materials than conventional application methods. GCP glass carbomer restorative material showed weaker SBS values than expected.

**Keywords:** Er,Cr:YSGG laser; Glass carbomer; Shear bond strength

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### Introduction

Besides advantages of the Glass Ionomer Cements (GICs) such as chemical bonding to enamel and dentin, fluoride releasing and no setting shrinkage, their moisture sensitivity and lack of physical strength are their critical disadvantages [1]. To improve the mechanical properties of glass ionomer; resin modified glass ionomer (RMGI) and polyacid modified resin (compomer) cements were introduced [2]. Due to the content of toxic (co)monomers (hydroxyethylmethacrylate (HEMA), triethyleneglycoldimethacrylate (TEGDMA), urethanedimethacrylate (UDMA) and bisglycidylmethacrylate (bisGMA)) of RMGI and compomer [3], a new glass ionomer based restorative material; glass carbomer cement (GCC) has been developed recently [4]. This new material contains nanosized powder particules and fluorapatite as secondary filler [5]. Its clinical application is similar with conventional glass ionomer cements, except that recommended heat application during setting reaction. Application of heat has been supposed to accelerate the matrix-forming reaction of glass carbomer [6]. GCCs are able to bond physico-chemically to enamel and dentin without any pretreatment of the surface [7]. Tooth surface treatment before the application of GIC has been reported to improve the bond strength [8]. Application of acidic conditioners is recommended to remove surface contaminants and the smear layer which might limit the bond of the GIC to tooth structure [9]. An alternative method of conditioning of tooth surface is

**Table 1:** Materials, manufacturers and chemical compositions of the specimens.

Material	Manufacturer	Chemical Composition
Fuji IX GP capsule	GC Corporation, Tokyo, Japan	Polyacrylic acid, fluoro-alumino-silicate glass, distilled water
Fuji II LC capsule	GC Corporation, Tokyo, Japan	HEMA <sup>+</sup> , UDMA <sup>+</sup> , polyacrylic acid, fluoro-alumina-silicate glass, distilled water
Dyract XP compule	Dentsply, Konstanz, Germany	UDMA <sup>+</sup> , dimethacrylate resin, strontium fluoride, butanedioic acid, strontium-flouro silicate glass
GCP capsule	GCP Dental, Ridderkerk, The Netherlands	Fluoro-alumino-silicate glass, polyacids
ETCH-37	Bisco Inc., Illinois, USA	37% phosphoric acid in H <sub>2</sub> O, thickeners, pigments
GC Cavity Conditioner	GC Corporation, Tokyo, Japan	20% polyacrylic acid, 3% aluminum chloride hexahydrate, distilled water
Prime&Bond NT	Dentsply, Konstanz, Germany	Di-Trimethacrylate resins, PENTA <sup>+</sup> , nanofillers-amorphous silicon dioxide, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone
GCP Gloss	GCP Dental, Ridderkerk, The Netherlands	Modified polysiloxanes
GC Fuji Coat LC	GC Corporation, Tokyo, Japan	Multifunctional urethane methacrylate, aliphatic methacrylate, methyl methacrylate

<sup>+</sup>Hydroxyethylmethacrylate <sup>+</sup>Urethanedimethacrylate <sup>+</sup>Dipentaerythritolpentacrylate phosphoric acid.

**Table 2:** Statistical analysis of restorative materials regarding surface conditions with "Post Hoc LCD" test.

Material	Surface Condition	P
CIS (Fuji IX)	control' acid	0.144
	control' laser	0.196
	acid' laser	<b>0.007</b>
RMGI (Fuji II)	control' acid	0.137
	control' laser	<b>0.000</b>
	acid' laser	<b>0.000</b>
Compomer (Dyract XP)	control' acid	<b>0.000</b>
	control' laser	<b>0.003</b>
	acid' laser	0.063
Glass Carbomer (GCP)	control' acid	0.470
	control' laser	0.493
	acid' laser	0.971

application of laser technology. Laser irradiated dentin surface has been shown very rough with irregularities and craters resembling the surface obtained by acid etching [10]. Thus, it is thought that laser conditioning might increase adhesion between restorative material and the tooth substrate. A new generation of erbium laser, Er,Cr:YSGG laser, can be used for conditioning of tooth substrate. The Er,Cr:YSGG laser has a wavelength 2,780 nm which uses laser energy, water and air. Irradiation with Er,Cr:YSGG laser produces a

rough dentin surface without smear layer, with open dentinal tubules and protruded peritubular dentin [11]. Conditioning tooth structure with Er,Cr:YSGG laser can be an appropriate alternative technique to etching 37% phosphoric acid for removing the smear layer and preparing dentin [12]. Increasing the use of laser in dentistry and improving the mechanical properties of GICs, the aim of this study is determined to evaluate the shear bond strengths of glass ionomer and glass carbomer cements on Er,Cr:YSGG laser or acid etched human dentin surface.

### Materials and Methods

One hundred and twenty extracted caries-free human third molar teeth were collected. Tissue debris and calculus were removed with periodontal curettes under running water. The teeth were stored in 0.5% chloramine T solution for one week and later in normal saline solution at room temperature. The teeth were embedded in self cured acrylic resin with help of silicon molds of 2.0 cm × 1.5 cm × 1.5 cm such that occlusal surfaces were parallel to acrylic resin block surface. Occlusal enamel was removed to obtain a flat surface of dentin. To simulate clinical conditions for removing enamel tissue, a standard high speed dental handpiece (NSK, Pana-Max, Nakanishi Inc., Japan) with 1 mm fissure bur (Komet, Lemgo, Germany) were used under water irrigation. All prepared specimens were then stored in distilled water for 24 h at room temperature and teeth were randomly divided into three groups. Each group consisted of forty teeth.

**Table 3:** The mean shear bond strengths and standard deviation for each group.

Material	Surface Condition	Mean (MPa)	n	Sd.	Min. (MPa)	Max. (MPa)
Fuji IX GP	Control	3.70	10	0.60	2.95	4.61
	Acid	4.34	10	0.71	3.08	5.67
	Laser	3.12	10	1.10	2.05	5.29
Fuji II LC	Control	5.43	10	0.10	3.92	6.72
	Acid	6.09	10	1.43	4.57	8.20
	Laser	3.75	10	0.97	2.38	5.44
Dyract XP	Control	6.54	10	0.80	4.82	7.24
	Acid	8.70	10	1.23	7.35	10.24
	Laser	7.88	10	1.65	6.28	10.07
GCP	Control	0.86	10	0.40	0.20	1.52
	Acid	1.18	10	0.28	0.65	1.42
	Laser	1.16	10	0.57	0.61	1.98

### Acid etched group

For Fuji IX GP (cGIC), Fuji II LC (RMGIC) and GCP (GCC), the occlusal dentin was conditioned with 20% polyacrylic acid (GC Cavity Conditioner, GC, Tokyo, Japan) for 10 s, washed with water for 10 s and gently air dried. The capsule of the Fuji IX, Fuji II and GCP were activated and mixed according to manufacturers' directions. The prepared mixture was applied into a cylindrical plastic mold (a diameter of 4 mm and a height of 4 mm) placed onto the treated dentin surface and condensed with the help of titanium-coated instrument and stainless steel condenser. Fuji II samples were cured with visible light source (Starlight S, Mectron s.p.a, Carrasco, Italy) in two increments for 20 s. GCP samples were cured with a high output range visible light source (GCP CarboLed Lamp, GCP Dental, Ridderkerk, The Netherlands) for 90 s (Table 1).

For Dyract XP (compomer), occlusal dentin was conditioned with 37% phosphoric acid gel (GCP CarboLed Lamp, GCP Dental, Ridderkerk, The Netherlands) for 15 s, washed with water for 10 s and gently air dried. Prime and Bond NT bonding agent was applied on to the surface and light cured for 10 s. The compule of the Dyract XP was applied into cylindrical plastic mold placed onto the treated dentin surface in two increments and condensed with the help of titanium-coated instrument and stainless steel condenser. Dyract samples were cured with visible light source for 40 s (Table 1).

### Laser etched group

The dentin surface was treated with Er,Cr:YSGG laser (Biolase Inc., San Clemente, CA) at a pulse energy of 0.5 W (65% air, 55% water) for 15 s with 600 micron diameter G-type sapphire tip. The laser tip was aligned perpendicular to the target area at a distance of 1 mm from the surface. This laser has a 2,780 nm wavelength with pulse duration of 140-200 micron s, a fixed 20 Hz repetition rate and average output power that could be adjusted from 0 W to 6 W. After laser conditioning of dentin surfaces, all restorative materials were placed into molds as the laser etched group.

### Control group

Neither acid nor laser was used for etching dentin surfaces. All restorative materials were applied into molds as the acid control group.

After removing the molds, a layer of gloss was applied to the all surfaces of each sample except Dyract XP specimens. All samples were then stored in artificial saliva at 37°C for 24 h. Shear bond strengths of the restorative materials were measured with Shimadzu AGS-X 50 kN testing machine at crosshead speed of 0.5 mm/min. The fractured surfaces were examined in stereomicroscope and SEM, categorized as:

1. Type 1: Adhesive failure between restorative material and dentin.
2. Type 2: Cohesive failure in dentin or restorative material.
3. Type 3: Mixed failure when cohesive failure and adhesive failure occurred in the same specimen.

All specimens were first examined under optical microscope to identify failure mode.

In order to prepare specimens for SEM analysis, they were gold-sputtered and exposed to high vacuum. Then the specimens were examined under a SEM (Jeol JSM-5910 LV, Jeol Ltd, Tokyo, Japan).

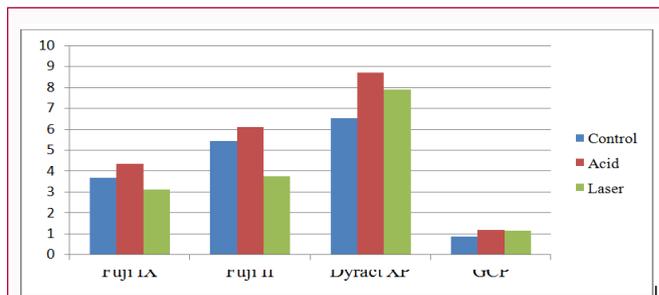


Figure 1: Mean shear bond strengths of each group (Mpa).

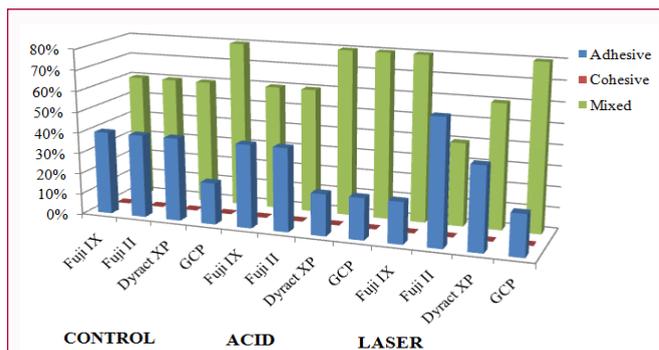


Figure 2: Fracture types percentages of the different groups tested.



Figure 3: Fuji IX control group mixed failure.

Statistical analysis was made using SPSS program for Windows 21.0 with Post Hoc LCD test (Table 2).

## Results

### Shear bond strength

The mean shear bond strengths and standard deviations for each group are shown in Figure 1 and Table 3. For Fuji IX groups, there were no statistical differences between acid etch and control groups and also laser etch and control groups ( $p > 0.05$ ). Statistical difference was observed between acid etch and laser etch groups ( $p < 0.05$ ). Bond strength values in acid etched group were higher than laser etched group. In Fuji II groups, there was no statistical difference between acid etch and control groups ( $p > 0.05$ ). The application of laser showed significantly lower bond strength than control and acid etch groups ( $p < 0.05$ ). For Dyract XP groups, acid and laser groups showed significantly higher bond strength values than control group ( $p < 0.05$ ). However, there is no significant differences between laser and acid applications ( $p > 0.05$ ). For GCP groups, there was no significant difference between in all conditions ( $p > 0.05$ ). In all different conditions of the dentin surface treatment, bond strength values were observed highest in Dyract XP groups and lowest in GCP groups ( $p < 0.05$ ).



Figure 4: Fuji IX acid group mixed failure.



Figure 8: Fuji II laser group mixed failure.

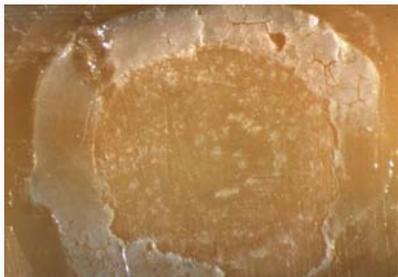


Figure 5: Fuji IX laser group adhesive failure.



Figure 9: Dyract XP control group adhesive failure.



Figure 6: Fuji II control group adhesive failure.



Figure 10: Dyract XP acid group mixed failure.



Figure 7: Fuji II acid group mixed failure.



Figure 11: Dyract XP laser group mixed failure.



Figure 12: GCP control group mixed failure.

**Fracture analysis**

Regarding the stereo microscope and SEM analysis, two types of failure modes were revealed; adhesive and mixed failure. The other failure type called cohesive failure was not observed. Mixed type of failure was observed more than adhesive type in all groups. Mixed type of failure percentages were 60% in control, 70% in acid and 65% in laser groups. SEM imaging showed that some restorative materials residue attached on the dentin surface in mixed failures (Figures 2-10). In SEM analyses, open dentinal tubules and absence of smear plugs was observed in the specimens conditioned with 37% phosphoric acid. And also open dentinal tubules were observed in the specimens conditioned with 20% polyacrylic acid but there were smear plugs



Figure 13: GCP acid group mixed failure.

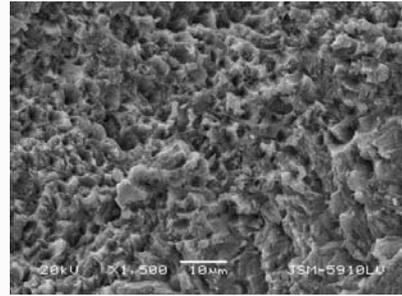


Figure 17: Er,Cr:YSGG laser (energy at 0.5 W) etched dentin surface.



Figure 14: GCP laser group adhesive failure.

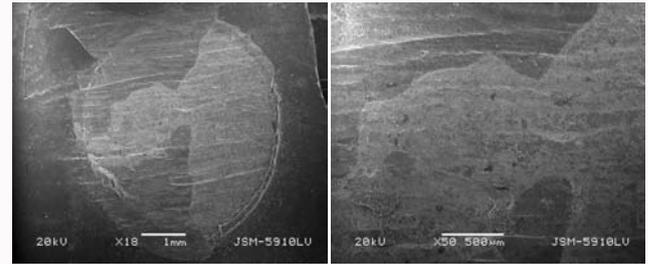


Figure 18: Fuji IX acid etch group - mixed failure.

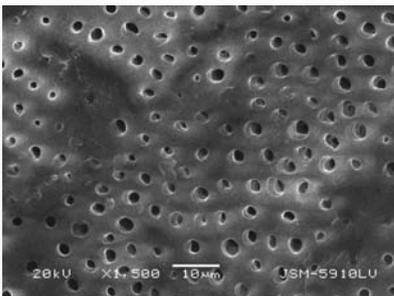


Figure 15: 37% phosphoric acid etched dentin surface.

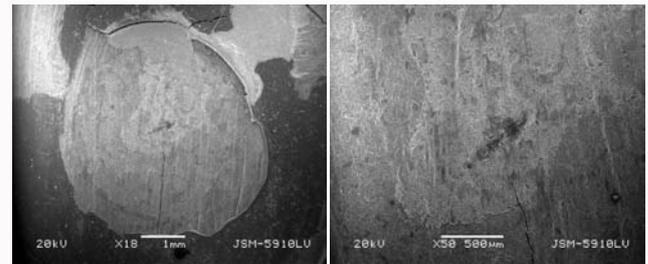


Figure 19: Fuji II laser etch group - mixed failure.

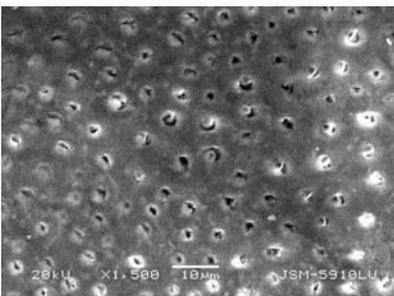


Figure 16: 20% polyacrylic acid etched dentin surface.

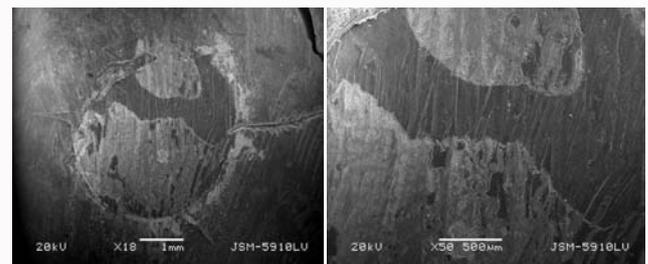


Figure 20: Dyract XP acid etch group - mixed failure.

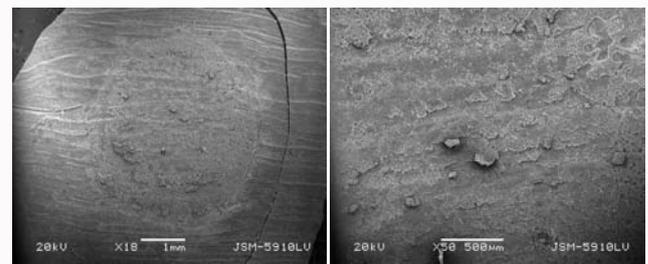


Figure 21: GCP control group - mixed failure.

in the tubules. For laser etched specimens irregular dentin surface pattern and some remnants of smear layer were observed Figures 11-21.

### Discussion

When using a bur to prepare tooth structure, a debris zone, smear layer develops on dentin surface [13,14]. This layer decreases surface wettability and causes weak adhesion between tooth and restoration [15]. To increase adhesion to tooth structure, it is advised that an acidic conditioner application onto tooth surface to remove

smear layer before tooth restoration [16,17]. Thirty seven percent of phosphoric acid gel for 15-20 s was applicator to enhance adhesion

of resins [18] and 25% polyacrylic acid solution was found to be quite effective to enhance adhesion of glass ionomer cements [19]. Glass ionomer based cements also bond to enamel and dentin, even with the presence of smear layer. However conditioners have been found to improve adhesion to tooth. It is reported that application of an acidic conditioner to dentin surfaces increased bond strength of glass ionomer cements [7,19,20-22] that the use of polyacrylic acid as a dentin conditioner removed smear layer and provided good wetting of the surface [19]. On the other hand, Hewlett et al. [23] reported that dentin conditioning with 25% polyacrylic acid reduced the affinity of the smear layer for dentin, leaving behind a poorly attached layer of smear debris, rather than removing it. Also Tyas [24,25] and Tanumiharja et al. [26] reported that the use of surface conditioners to improve the bond strength of glass ionomer cements was not necessary. In this study, conditioning dentin surface with 20% polyacrylic acid produced no difference in bond strengths of conventional glass ionomer, resin modified glass ionomer and glass carbomer restorative materials. According to the manufacturers of Dyract XP material (compomer), it is reported that acid etching improved the bond strength of the material, and also it is reported that dentin conditioning was not necessary. Yap et al. [27] reported that one of the unique features of the compomer's adhesion to tooth structures was the omission of acid etching. And also the authors reported that the material of compomer was self-adhesive because of the hydrophilic carboxylic groups presented in its patented TCB (reaction product between butane tetracarboxylic acid and hydroxyethyl methacrylate) resin [27]. In the literature there are so many studies that acid etching improved the bond strength of compomers [28-30] which was confirmed in the present study. To remove smear layer and increase adhesion of restorations, conditioning tooth structure with Er,Cr:YSGG laser can be an appropriate alternative technique to etching with 37% phosphoric acid. Some researchers reported that Er,Cr:YSGG laser with water cooling was effective for removing caries and etching tooth substrate [31-33]. It was showed that using Er,Cr:YSGG laser which removes smear layer and opens dentinal tubules increased the bond strength of the restorations [33,34]. In recent studies, Garbui et al. [35] reported that conditioning with Er,Cr:YSGG laser with average power of 0.5 W, 25 mJ per pulse and energy density of 9 J/cm<sup>2</sup> increased bond strengths between cGIC and dentin. However, Ekworapoj et al. [36] reported that Er,Cr:YSGG laser conditioning did not increase bond strength of GIC to dentin and also Jordehi et al. [37] reported that Er,Cr:YSGG laser conditioning (at energy 1 W) did not improve bond strength of Fuji II LC to dentin and even decreased bond strength of Fuji II LC [36,37]. In the present study, similar with the recent studies [36,37] bond strength of GICs to dentin did not increase significantly with Er,Cr:YSGG laser conditioning and also laser application decreased bond strength of Fuji II LC to dentin. Although some studies reported that laser conditioning improved the bond strength of restorative materials due to the absence of smear layer, open dentinal tubules and irregular tooth surface, transmission electron microscope evaluations showed no-identifiable, fused collagen fibrils and lack of interfibrillar collagen space on the laser treated dentin. Consequently it is reported that lack of interfibrillar collagen space was responsible for inhibition of resin diffusion into dentin surface and lower bond strength values with Fuji II LC [37]. In this study, Dyract XP showed the highest bond strength values in all dentin surface treatments. It may be considered that both chemical and micro-mechanical bonding of Dyract XP provided the highest bond strength values. Moreover, bond strength values of Fuji II were

found higher than Fuji IX. It was considered that the presence of HEMA which provides wetting ability and penetration to exposed collagen network in Fuji II [38] caused better bond strength values. GCP showed the lowest bond strength values in all dentin surface treatments. There was no significant difference in all dentin surface conditions for GCP. In the literature, there is no published data about the bond strength of glass carbomer restorative material. In vitro tests may provide valuable insights into mechanical properties of this new material [39,40].

## Conclusion

Based on these findings, it may be concluded that:

- 37% phosphoric acid conditioning improved the bond strength of compomer.
- 20% polyacrylic acid and laser conditioning (energy at 0.5 W) did not increase bond strength of cGIC, RMGI and GC restorative materials and even reduced bond strength of RMGI (Fuji II LC).
- Glass carbomer restorative material showed significantly lower bond strength values to dentin than other restorative materials evaluated in this study.

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