

Comparison of the Shear Bond Strength between a Self-Etching Ceramic Primer and Conventional Bonding Systems Used in Bonding Metal Brackets to a Ceramic Surface

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ABSTRACT

Introduction: The purpose of this study was to compare the shear bond strength (SBS) of metal brackets bonded to ceramics with a self-etching ceramic primer (Monobond Etch & Prime™) versus other bonding systems, and to evaluate changes to the ceramic surface with bonding. **Methods:** Fifty ceramic discs were divided into five groups (n=10); After Group A-D were etched with 9.5% hydrofluoric acid (HF), they were pre-treated with Monobond N™ (Group A), silane (Group B and C), and Scotch-bond Universal Adhesive™ (Group D). Group E was pre-treated with Monobond Etch & Prime™ (MEP) but without etching. Transbond XT Light Cure Primer™ was applied to all groups except for Group C, in which Assure Plus™ was used instead. SBS was measured with a universal testing machine. Surface roughness and topography of the ceramic surfaces conditioned with an HF only, an HF and silane, and MEP was evaluated using a confocal laser scanning microscope and a scanning electron microscope. **Results:** Group E achieved a clinically adequate SBS. Group D showed significantly higher SBS than Groups A and E ($P<0.05$). Group C showed higher SBS than Group A ($P<0.05$). **Conclusions:** The ceramic surfaces conditioned with MEP showed less surface roughness and etching pattern than those etched with HF. The MEP showed appropriate SBS like those seen with other bonding systems, but there was less ceramic surface damage than on those treated with HF. (Clin J Korean Assoc Orthod 2024;14(2):81-89)

Key words Self-etching ceramic primer, Shear bond strength, Surface roughness



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INTRODUCTION

With the increase in adults seeking orthodontic treatment, orthodontists are no longer just dealing with natural dentitions but also with various dental restorations. For an efficient and effective treatment process, adequate materials and techniques are required to establish proper bonds on surfaces consisting of various types of restorative materials.¹ If there are repeated bonding failures during treatment, the treatment time may be longer. According to Reynolds,² it is estimated that orthodontic brackets need a shear bond strength (SBS) range of 5.9 to 7.8 MPa to withstand masticatory forces. Many articles cite that this SBS range provides clinically acceptable values and that current bonding systems meet or exceed this requirement. For example, it is reported that metal brackets bonded to enamel with a conventional 2-step etch and Transbond XT composite resinTM (3M Unitek, Monrovia, Calif) have a bond strength of 20.2 MPa.³

Another consideration is the potential damage to the enamel or restoration surface associated with bracket removal; the removal of brackets should be kept to a minimum.⁴ If a cohesive failure occurs in the enamel or restorations, it may cause cracks or tear-outs on the surfaces.⁵ Therefore, the range of bonding strength should be suitable for bracket bonding.

Since ceramics are chemically inert and interact poorly with possible reactants, bonding brackets to a ceramic surface cannot be achieved with commonly used adhesive systems. A chemical and/or mechanical pre-treatment of the ceramic surface is required to improve the adhesive bond between ceramic and the bracket base. Numerous pre-treatment methods have been suggested including mechanical roughening the surface with a bur, laser, or sandblasting, or chemically conditioning the surface by etching it with hydrofluoric acid or phosphoric acid. Although Schmage et al.⁶ was unable to determine an ideal surface conditioning method for bonding metal brackets to ceramic surfaces, they added that the use of hydrofluoric acid would still be appropriate. Since other methods like sandblasting and silane applications unnecessarily increased the surface roughness, ceramic damage was observed during debonding after these processes. Unfortunately, intraoral application of hydrofluoric acid is not recommended, since it is hazardous to soft tissues and can even cause bone necrosis.^{4,7}

To eliminate the risk factors with hydrofluoric acid, Monobond Etch & PrimeTM (MEP; Ivoclar Vivadent,

Liechtenstein, Germany), was introduced to the market recently. It is a self-etching ceramic primer that combines hydrofluoric acid etching with a silanization procedure. MEP contains a less aggressive acid (ammonium polyfluoride) for etching along with a silane system (trimethoxypropyl methacrylate) for silanization. Therefore, MEP reduces the conventional two-step ceramic conditioning procedure to one step. Importantly, according to the manufacturer, MEP generates a less rough surface than hydrofluoric acid.⁸ In previous studies investigating bond strength between CAD-CAM ceramics⁹ or glass ceramics⁸ and resin composite luting cement, the bond strength of MEP was comparable to other conventional bonding systems. In contrast, in the study of El-Damhoury and Gaintantzopoulou,¹⁰ MEP showed significantly lower SBS values compared to hydrofluoric acid and silane. For this reason, they suggested that hydrofluoric acid and silane are the gold standard for pretreatment of ceramics for resin-luting cementation.

The aim of this study was to compare the shear bond strength of a new self-etching primer-Monobond Etch & PrimeTM (MEP) and conventional bonding systems used in bonding metal brackets to ceramic surfaces.

MATERIALS AND METHODS

Specimen preparation

Ceramic blocks (Vita omega 900TM; VITA Zahnfabrik, Bad Säckingen, Germany) were formed into rectangular sections (882 mm³). All specimens were positioned in polyvinyl chloride (PVC) plastic rings (height 48 mm, diameter 38 mm) and were embedded in hard stone (Rhombstone WhiteTM; Ryoka Dental, Mie-Ken, Japan). Polishing was performed with a ceramic polishing bur (PressPolTM; EVE, Florida, USA) until the ceramic surface looked shiny to the naked eye, simulating clinical procedures. After the polishing, the samples were cleaned and dried with ethyl alcohol.^{10,11} All 50 specimens were randomly divided into 5 groups (n=10).

The surfaces of Groups A-D were etched with 9% hydrofluoric acid (Ultradent Porcelain etchTM; Ultradent, Utah, USA), with an etching time of 90 seconds according to the manufacturer's instructions. Surfaces were rinsed thoroughly for 20 seconds and then dried for 10 seconds.

Next, Monobond NTM (Ivoclar Vivadent, Liechtenstein, Germany) was applied in a thin layer to the samples in Group A for 60 seconds and was then dried for

60 seconds. Ultradent Porcelain Silane™ (Ultradent, Utah, USA) was applied and dried in the same way as the samples in Groups B and C. For Group D, Scotchbond Universal Adhesive™ (3M ESPE, Minnesota, USA) was used. After application for 20 seconds, it was light cured for 10 seconds. For all light curing, VALO™ (Ultradent, Utah, USA) was applied parallel to the surface at a distance of 1 cm.

MEP, a self-etching ceramic primer was applied to Group E. After agitating it on the ceramic surface for 20 seconds with a micro brush, it was left for an additional 40 seconds to react on the ceramic surface. After the reaction time, the ceramic surface was rinsed with a water spray for 20 seconds and was then dried for 10 seconds.

After the application of the ceramic primer, Transbond XT Light Cure Primer™ (3M Unitek, California, USA) was applied to all groups except for Group C and was light cured for 10 seconds. With Group C, Assure PLUS All Surface Bonding Resin™ (Reliance, Sydney, Australia) was used instead.

For the last procedure, Transbond XT Light Cure Ad-

hesive™ (3M Unitek, California, USA) was applied to the bracket base, which was affixed to the ceramic sample and light cured for 10 seconds. Upper central incisor metal brackets (Mini master brackets™; American orthodontics, Wisconsin, USA) were used in this study. The average contact area of the bracket base was 10.5 mm². All these procedures were performed by the same operator. All materials and protocols used in this study are shown in Tables 1 and 2.

Shear bond strength

The shear bond strength (SBS) was measured with a universal testing machine (WL2001™; Withlab, Gyeonggi, Korea) with a 3000 N load cell. The velocity of the force introduced was 1 mm/min. The load at bracket failure was measured in newtons (N),¹² and the force (N) was divided by the surface area of the bonding site (mm²), therefore the SBS was expressed in MPa.

Surface roughness measurement

Three randomly chosen ceramic specimens were treat-

Table 1. Manufacturers and composition of materials used in the study

Material	Manufacturer	Composition
Ultradent Porcelain Etch	Ultradent, Utah, USA	9.5% buffered hydrofluoric acid solution
Ultradent Porcelain Silane	Ultradent, Utah, USA	Methacryloxy propyl trimethoxy silane, isopropyl alcohol
Monobond Etch & Prime	Ivoclar Vivadent, Liechtenstein, Germany	Tetrabutylammonium-dihydrogentrifluoride, silane system (based on trimethoxypropyl methacrylate), methacrylate phosphoric ester, butanol, water, food coloring (Fast Green)
Monobond N	Ivoclar Vivadent, Liechtenstein, Germany	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulfide methacrylate
Scotchbond Universal Adhesive	3M ESPE, Minnesota, USA	Bis-GMA, HEMA, decamethyldimethacrylate, 10-MDP, Vitrebond Copolymer, filler, ethanol, water, initiators, silane
Assure Plus	Reliance, Sydney, Australia	HEMA, MDP and Bis-GMA

Bis-GMA: bisphenol A-glycidyl methacrylate; HEMA: hydroxyethylmethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

Table 2. Application procedures done in each group

Group	Etching	Ceramic primer	Adhesive	Light cure resin
A	9.5% hydrofluoric acid	Monobond N	Transbond XT, Light Cure Primer	Transbond XT, Light Cure Adhesive
B	9.5% hydrofluoric acid	Ultradent Porcelain Silane	Transbond XT, Light Cure Primer	Transbond XT, Light Cure Adhesive
C	9.5% hydrofluoric acid	Ultradent Porcelain Silane	Assure Plus	Transbond XT, Light Cure Adhesive
D	9.5% hydrofluoric acid	Scotchbond Universal Adhesive	Transbond XT, Light Cure Primer	Transbond XT, Light Cure Adhesive
E	Monobond Etch & Prime		Transbond XT, Light Cure Primer	Transbond XT, Light Cure Adhesive

ed respectively as follows to observe the change of the ceramic surface; 9.5% hydrofluoric acid only, 9.5% hydrofluoric acid followed by a silane application, and MEP only. After being rinsed with distilled water, all specimens were immersed in an ultrasonic water bath for 3 minutes and were dried with oil-free air for 20 seconds.

The surface topography was studied with a confocal laser scanning microscope (CLSM) (LSM700™; Zeiss, Oberkochen, Germany). A reflected image of the surface was generated by an Ar/Ar Kr laser, with a scan format of 512 × 512 pixels and scan speed of 400 Hz. The z-series generated by the light was converted to a greyscale (topographical) image which was used to measure the surface roughness. The three-dimensional topographical resolution was achieved by using ZEN 2009 software (Zeiss, Jena, Germany). The average surface roughness (Ra), peak height (highest value; Rp), valley depth (lowest value; Rv), root mean square roughness (Rq) and maximum roughness height (Rt) of the specimens were calculated as a numeric value in micrometers.¹⁰

Surface topography examination

To observe the topography of the ceramic surface after surface conditioning, three specimens were randomly selected again after being processed with the protocols mentioned above. After being air-dried for 60 s, specimens were mounted on aluminum stubs and were sputter coated with 100Å of Gold-Palladium (EMS 7620 Mini Sputter Coater™, Pennsylvania, USA). The surface topography of each sample was examined with a scanning electron microscope (EM-30™, COXEM, Daejeon, Korea) operating at 20 kV acceleration voltage and 15 mm working distance.

Statistical analysis

IBM SPSS Statistics 25.0 software was used for statistical analysis. SBS means and standard deviations were

calculated. Normal distribution of the data for each group was confirmed using the Kolmogorov-Smirnov test & Shapiro-Wilk test. Analysis of variance (ANOVA) and Tukey HSD (honestly significant difference) test was done to determine the significant differences between the groups. The significance level was set at $P < 0.05$.

RESULTS

Shear bond strength

Mean SBS values are shown in Table 3 and Figure 1. Means with the same letter indicate that there was no significant difference. SBS with Scotchbond Universal Adhesive™ (Group D) was significantly higher than with Monobond N™ (Group A) and Monobond Etch & Prime™ (Group E). Also, SBS was higher with Ultradent Porcelain Silane™ (Group C) than with Monobond N™ (Group A). All other groups resulted in similar mean SBS. Scotchbond Universal Adhesive™ (Group D) showed the highest mean value and Monobond N™ (Group A) showed the lowest mean value. Among all 50 samples, the lowest value was 4.6 MPa, with Monobond N™ (Group A) and the highest was 10.9 MPa, with Scotchbond Universal Adhesive™ (Group D).

Surface roughness measurements

The surface roughness test results are described in Tables 4, 5 and Figure 2.

Hydrofluoric acid etching showed a higher surface roughness value (Ra) than etching with MEP. Surface roughness decreased when silane was applied after hydrofluoric acid etching. Also, hydrofluoric acid etching showed the highest peak, deepest valley, and largest peak-valley height value. MEP was the next highest and silane application was the lowest. These results were visually confirmed and are illustrated in Figure 3. Hydrofluoric acid etching shows more surface irregularities

Table 3. Descriptive statistics of the SBS value in all groups

Group	Primer	Adhesive	Mean SBS ± SD (MPa)
A	Monobond N	Transbond XT, Light Cure Primer	6.5±1.3
B	Ultradent, Porcelain Silane	Transbond XT, Light Cure Primer	8.4±0.7
C	Ultradent, Porcelain Silane	Assure Plus	9.2±1.1
D	Scotchbond, Universal Adhesive	Transbond XT, Light Cure Primer	9.8±1.0
E	Monobond Etch & Prime	Transbond XT, Light Cure Primer	7.5±0.6

SBS: shear bond strength.

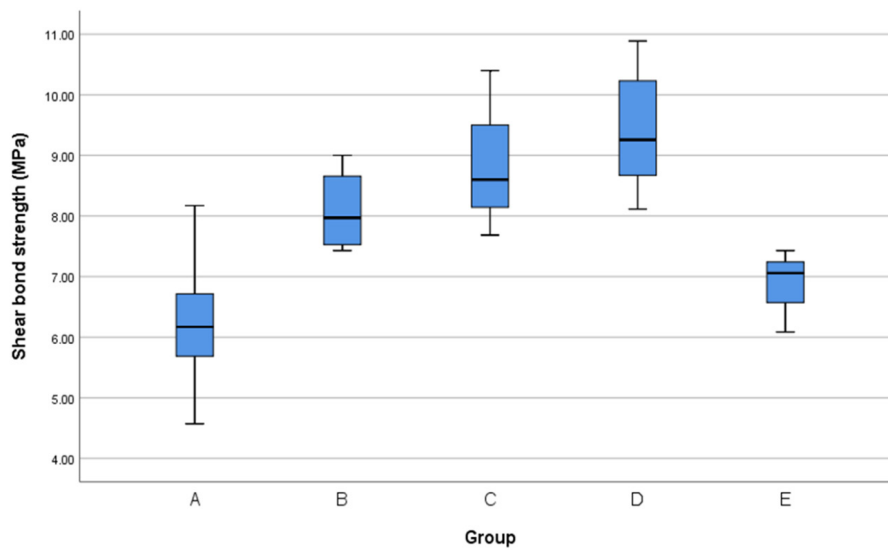


Figure 1. Box-plot of 95% confidence interval measurements and mean values of shear bond strength in all groups.

Table 4. Comparison of shear bond strength (MPa) between groups

Group A	Group B	Group C	Group D	Group E
a	a			a
	b	b	b	b
			e	

ANOVA and Tukey HSD test showed that the same letter was not significantly different.

Table 5. Roughness after conditioning of the ceramic surface

Pre-treatment	Ra (μm)	Rp (μm)	Rv (μm)	Rq (μm)	Rt (μm)
HF	3.554	9.332	9.922	4.311	19.255
HF + S	1.191	3.594	4.620	1.516	8.214
MEP	1.516	5.537	5.779	1.882	11.316

Ra: surface roughness value; Rp: highest peak value; Rv: lowest valley value; Rq: root mean square roughness; Rt: maximum roughness height; HF: hydrofluoric acid; HF+S: hydrofluoric acid and silane; MEP: Monobond Etch & Prime.

and pores compared to MEP etching.

Surface topography examination

SEM images of the treated ceramic surfaces are presented in Figure 4. Irregular surfaces with more pores and defects were seen in hydrofluoric acid etching than with MEP. The ceramic surface conditioned with hydrofluoric acid and silane showed similar irregularities as those with the sample that was treated with only hydrofluoric acid.

DISCUSSION

Since the ceramic structure is inert, various techniques are required when bonding a bracket on a ceramic surface to increase the bond strength.¹³ These processes aim to change the ceramic surface either mechanically or chemically. Hydrofluoric acid etching of feldspathic and lithium disilicate ceramics, combined with a silane coupling agent has been considered as the gold standard for the treatment of the silica-based ceramics.¹⁴ In a recent systematic review, the best protocol suggested by the au-

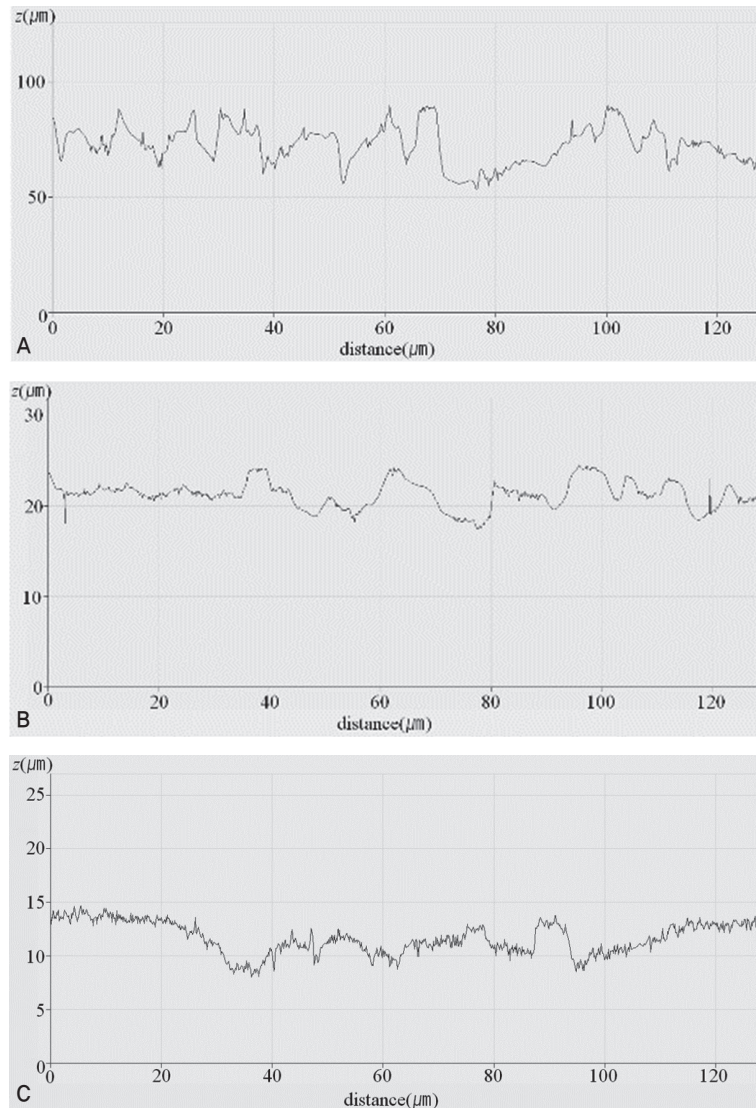


Figure 2. Mean roughness values of each ceramic surface after conditioned with hydrofluoric acid (A); hydrofluoric acid and silane (B); and Monobond Etch & Prime (C).

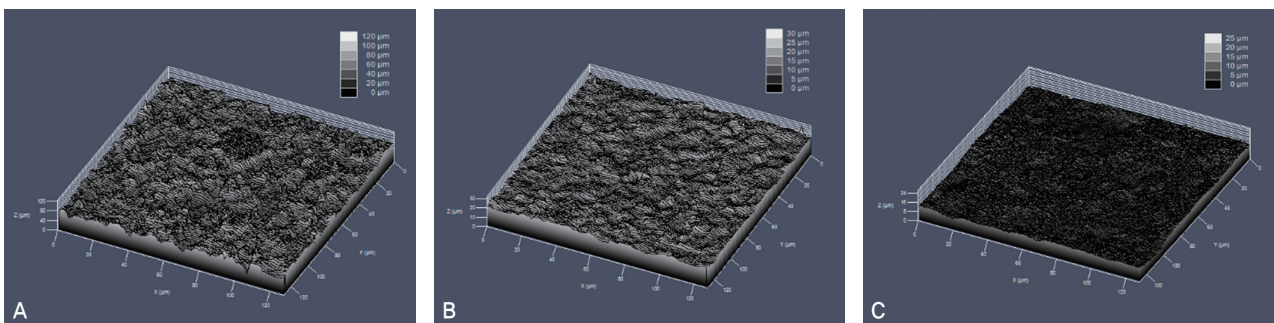


Figure 3. Confocal laser scanning micrograph images of the ceramic surface after conditioned with hydrofluoric acid etching (A); hydrofluoric acid etching and silane (B); and Monobond Etch & Prime (C).

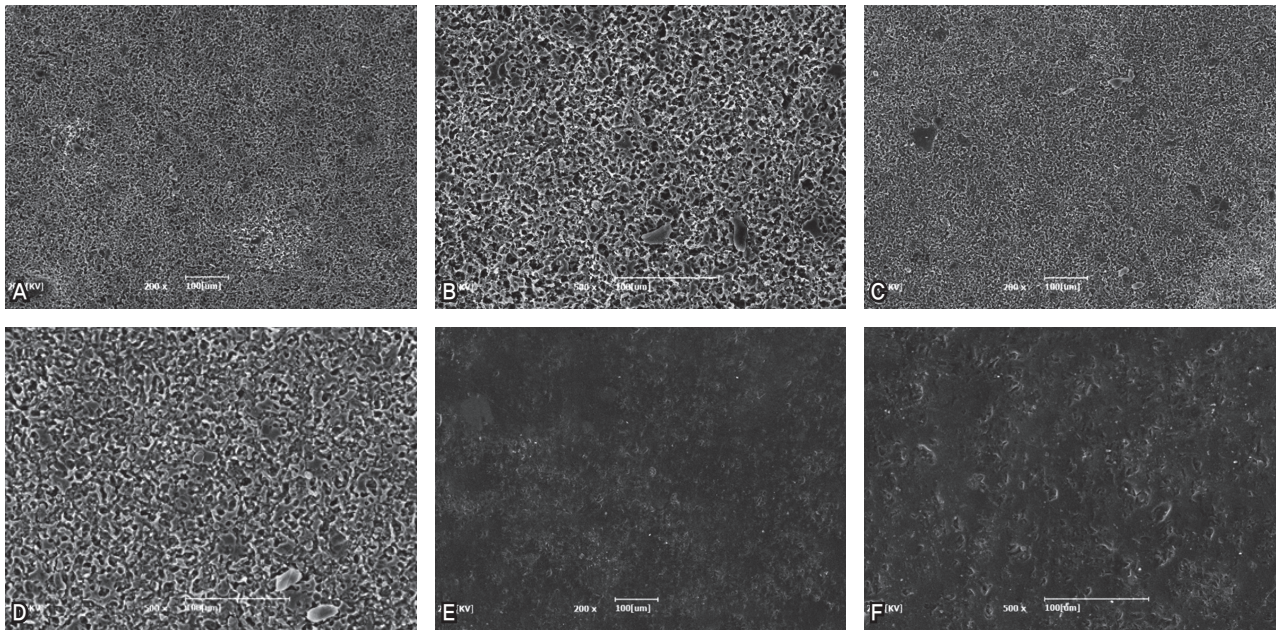


Figure 4. A, SEM images of the ceramic surface conditioned with hydrofluoric acid ($\times 200$). B, SEM images of the ceramic surface conditioned with hydrofluoric acid ($\times 500$). C, Hydrofluoric acid etching and silane ($\times 200$). D, Hydrofluoric acid etching and silane ($\times 500$). E, Monobond Etch & Prime ($\times 200$). F, Monobond Etch & Prime ($\times 500$) (magnification).

thors was 9.6% hydrofluoric acid etching for 1 minute followed by the application of silane.¹⁵

Hydrofluoric acid etching remarkably increases bond strength because of its reaction with the silica phase, inducing micromechanical retention through microchannels.¹⁵ However, it has disadvantages, too. First, the ceramic surface loses its glaze, and it becomes difficult for the clinician to restore it to its original luster.¹³ Second, hydrofluoric acid is hazardous to soft tissues.^{4,7} And the silane reacts with the resin and forms a bridge between resin and ceramic.¹³ A study that focused on analyzing ceramic surfaces treated with silane concluded that the bond strength of brackets to ceramic surfaces was improved by applying silane.¹⁶ Therefore, all primers and adhesives used in this study were selected because they contain silane.

Monobond NTM includes silane, phosphate functional monomer and sulfide functional monomer, which forms chemical bonds between resin and silica, non-noble metals, or noble metals. Assure PlusTM contains HEMA, MDP and Bis-GMA. And Bis-GMA is a large polymer with a molecular weight of 512 g/mol, which gives Assure PlusTM a higher viscosity.¹⁷ MEP includes ammonium polyfluoride for etching and trimethoxypropyl methacrylate for silanization.⁹ Scotchbond UniversalTM

is a universal adhesive which also contains silane.

Clinically adequate bond strength for a metal orthodontic bracket-to-enamel bond should range from 6 to 8 MPa.¹⁸ All mean SBS values in this study were above this optimal range, but there were SBS values below 6 MPa in Group A. Group D showed significantly higher SBS compared to Groups A and E while the highest bond strength value was in Group D.

Also, bonding failures after debonding occurred at the bracket base-resin interface, with all the resin remnant remaining on the surface in all groups except for Group D, which showed ceramic fracture on the surface of three specimens. This means that first, the bond between the adhesive resin and the ceramics was stronger than the mechanical locking attained by the bracket base, and secondly, the bond strength was greater than the cohesive strength of the luting resin. However, the cohesive failure on the ceramic surface in Group D implies that the ceramic-resin bond strength was higher than the ceramic itself. Considering that the failure pattern with composite resin bonded to ceramic generally becomes a cohesive failure in ceramic when bond strengths are higher than 13 MPa,^{18,19} the cohesive failures in Group D might have been induced by the high bond strength.

The MEP group (Group E) showed good SBS values be-

tween 6 to 8 Mpa and there was no significant difference compared to the conventional bracket bonding method (Group B). Some recent studies reported similar results in the bond strength with composite resin cement to ceramics between MEP and hydrofluoric acid etching.^{8,9,20-22} However, it was also reported that the MEP group showed statistically lower bond strength than the hydrofluoric acid etching groups.^{10,23,24} Lopes et al.²³ pointed out that these opposite results might have occurred because of different polishing methods used on ceramic specimens. In our study, as the polishing procedure was operated manually, this could have resulted in a less uniform surface condition. Most of the reported studies related to MEP focus on the bond strength between ceramics and resin luting cement. And there was only one study about the bracket bond strength to zirconia ceramics. Although they showed similar results with our study, they used yttrium stabilized zirconium oxide ceramics, Light Bond™ (Reliance, Illinois, USA) instead of glass ceramics and Transbond XT Light Cure Adhesive™. Also, there was no control group for comparison.⁴

Although the exact mechanism of MEP is not clear, MEP contains ammonium polyfluoride, which etches glass and related silicates to achieve a micro-mechanical retention. Since ammonium polyfluoride has a lower acidity than hydrofluoric acid, MEP causes less damage to the surface and is less dangerous.²⁵ This was confirmed by confocal laser scanning micrographs and SEM images. MEP resulted in a less porous and smoother surface than hydrofluoric acid. MEP also contains functional monomers such as methacrylate phosphoric acid ester (10-MDP), which increase potential chemical interaction in addition to silane.²⁵ These chemical components might be the reason why MEP shows clinically sufficient bond strength even though the surface of the ceramic is less porous.

One of the limitations of this study was that it was done *in vitro*; the bond strength could be different in actual clinical practice. Since the intraoral environment cannot be totally controlled as it was in our study, an *in vivo* study should be done to see the actual success rates with each bonding system.

CONCLUSIONS

The new self-etching ceramic primer Monobond Etch & Prime™ showed appropriate shear bond strength when used to bond a metal bracket to a ceramic sub-

strate. It showed similar bond strength with other bonding systems while causing less ceramic surface damage than hydrofluoric acid. It could be a positive alternative for metal bracket bonding to ceramic surfaces such as those used on dental restorations.

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