The effect of different ceramic surface treatments on the repair bond strength of resin composite to lithium disilicate ceramic

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This study aimed to evaluate the bonding abilities and long-term durability of different repair agents when applied to lithium disilicate ceramics (LDS). Blocks of IPS e.max CAD were prepared and divided into four groups according to the surface treatment: Monobond Etch & Prime (ME), K-etchant GEL+Clearfil Universal Bond (UB), Bondmer Lightless (BL), and K-etchant GEL+G-Multi Primer (GMP). All treated ceramic specimens were bonded to resin composite and light-cured. The micro-shear bond strength was measured after 24 h of water storage or 5,000 thermocycles. ME and BL showed significantly higher initial bond strengths than UB and GMP (p<0.05). After 5,000 thermocycles, there was no significant difference in ME (p>0.05), but BL and GMP showed considerable reduction in bond strength (p<0.05); moreover, the samples of UB were all de-bonded before testing. ME and BL proved to be effective surface treatment materials for LDS.

Keywords: Lithium disilicate ceramic, Repair, Micro-shear bond strength, Silane, Thermocycling

INTRODUCTION

Recently, all-ceramic restorations became popular due to an increase in the demand for aesthetic materials. Compared to metal, all-ceramic materials have multiple advantages such as translucency, fluorescence, and biocompatibility1-3. Dentists are provided with various options when selecting a material for single-unit crown fabrication. Lithium disilicate ceramics (LDS), which consist of a lithium oxide (Li2O) contained within a silica glass matrix, are one of the most frequently used materials for anterior crowns owing to their aesthetic optical property4. However, LDS is inferior to the highly translucent zirconia in terms of strength5. Once LDS is fractured intra-orally, total replacement of the fractured restorations compromises the intact tooth structure with increased preparation and restoration size. The FDI supports the management of dental caries that utilizes the principles of minimal intervention dentistry (MID), including the repair of defective restorations rather than their replacement6. Therefore, direct repair using a composite resin is considered another choice due to the preservation of supporting structures7-8.

Conventionally, hydrofluoric acid (HF) etching followed by silane-containing primer was a well-accepted and recommended method to obtain durable bonding to LDS because HF increased the roughness of the ceramic surface and consequently produced microporosities and undercut to achieve micromechanical retention9-11. However, HF is a highly corrosive material and can be absorbed into the blood and bone through the skin, which may lead to health issues like cardiac arrest12,13. Therefore, HF cannot be used in the oral cavity, and phosphoric acid (PA) is often utilized as a substitute. However, PA neither creates adequate mechanical retention nor removes silicon oil contamination. The effectiveness of PA etching on bonding to glass-ceramics has been controversial14-17. Sandblasting is another repair system of ceramics18, but it includes the risk of injuring the surrounding soft tissues and spreading particles of aluminum oxide over the operated area19.

A self-etching primer for glass-ceramic (Monobond Etch & Prime, Ivoclar Vivadent, Schaan, Liechtenstein) was recently developed to replace HF for the conditioning of ceramic surfaces and was reported to be effective as a pretreatment of LDS. Monobond Etch & Prime (ME) contains a silane (trimethoxysilylpropyl methacrylate), an etching-agent for ceramic (tetrabutyl ammonium dihydrogen trifluoroide), and a priming agent (methacrylated phosphoric acid ester) in a bottle, which enables surface etching coupled with silanization in one step20. Several studies revealed that ME had comparable bond strength to HF etching followed by the silanization of LDS, depending on the resin cements10,20,21. Although ME is much less hazardous than HF, it cannot be used in the oral cavity due to its slight cytotoxicity22.

The concept of universal adhesives introduces a simplified protocol for the surface treatment of ceramics. They are of either one-bottle or two-bottle type and contain silane and phosphate monomers that

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support the adhesion between ceramics and resin-based materials\textsuperscript{23,24}. Recent studies have shown that the incorporation of silane in a one-bottle universal adhesive does not result in the same bonding performance as in the case of a separately applied silane agent\textsuperscript{24-27}. Since only a few studies have examined the two-bottle type universal adhesives, further research should be conducted to evaluate new protocols of using these adhesives with cementation.

This study aimed to evaluate the bonding abilities and long-term durability of different repair agents to LDS. The null hypothesis tested was that the bonding performance and durability of LDS to resin composite would not be affected by different surface treatments.

### Table 1 Composition of materials used in this study

| Material                  | Manufacturer                  | Composition                                                                 | Application instructions | Batch No. |
|---------------------------|-------------------------------|-----------------------------------------------------------------------------|--------------------------|-----------|
| IPS e.max CAD             | Ivoclar Vivadent, Schaan, Liechtenstein | SiO$_2$, Li$_2$O, K$_2$O, Al$_2$O$_3$, P$_2$O$_5$, ZrO$_2$, ZnO, MgO, coloring oxides | —                        | U24880    |
| Monobond Etch & Prime (ME) | Ivoclar Vivadent             | butanol, trimethoxysilylpropyl methacrylate, bis (trialkoxysilyl) ethane, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, colorant | Applied, rubbed for 20 s, and left for a further 40 s for sufficient reaction. Washed off with water and air-dried for 10 s. | U391834   |
| Clearfil Universal Bond (UB) | Kuraray Noritake Dental, Tokyo, Japan | MDP, Bis-GMA, Hydrophilic aliphatic dimethacrylate, HEMA, colloidal silica, silane coupling agent, di-camphorquinone, ethanol, water | Applied for 20 s, air-dried with medium air pressure for 10 s, and light-cured for 10 s. | 2P0036    |
| Bondmer Lightless (BL)    | Tokuyama Dental, Tokyo, Japan  | A: MTU-6, Bis-GMA, TEGDMA, HEMA, acetone, phosphate monomer, others B: γ-MPTES, acetone, isopropanol, water, borate type catalyst, peroxide | Applied and immediately air-dried with medium air pressure for 10 s. | 004       |
| G-Multi Primer (GMP)      | GC, Tokyo, Japan              | MDP, MDTP, γ-MPTS, methacrylate monomer, ethanol                           | Applied for 20 s, and air-dried for 10 s. | 1702231   |
| K-etchant GEL             | Kuraray Noritake Dental       | phosphoric acid, colloidal silica, water, dyes                             | Applied for 15 s, flushed by water, and gently air-dried. | 8N0067    |
| Clearfil AP-X A2          | Kuraray Noritake Dental       | Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, camphorquinone, initiators, accelerators, pigments | —                        | 460070    |

\textsuperscript{23}Al$_2$O$_3$, aluminum oxide; Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; K$_2$O, potassium oxide; Li$_2$O, lithium superoxide; MgO, magnesium oxide; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MDTP, 10-methacryloyloxydecyl dihydrogen thiophosphate; MTU-6, 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; P$_2$O$_5$, phosphorus pentoxide; SiO$_2$, silicon dioxide; TEGDMA, triethylene glycol dimethacrylate; ZnO, zinc oxide; ZrO$_2$, zirconium oxide; γ-MPTES, γ-methacryloxypropyl triethoxy silane; γ-MPTS, γ-methacryloxypropyl trimethoxy silane

### MATERIALS AND METHODS

The materials and instructions used in this study are listed in Table 1. LDS (IPS e.max CAD, Ivoclar Vivadent) blocks were used in this study. A total of sixty-one LDS specimens (2 mm thickness×15 mm width×15 mm length) were obtained from the original LDS blocks using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). The LDS specimens were submitted to the crystallization-firing process in a furnace (Programat P300, Ivoclar Vivadent) at 840–850°C for 30 min according to the manufacturer’s recommendation.

**Micro-shear bond strength test**

A total of twenty-four LDS specimens were ground up to 1000-grit silicon carbide (SiC) paper under running water and then cleaned in distilled water using ultrasonicator for 3 min followed by air-drying. The
specimens were randomly allocated into the following four groups according to the treatment. Therefore six LDS specimens were used for each treatment.

1) Monobond Etch & Prime (Ivoclar Vivadent): ME
2) K-etchant GEL (Kuraray Noritake Dental) + Clearfil Universal Bond (Kuraray Noritake Dental): UB
3) Bondmer Lightless (Tokuyama Dental, Tokyo, Japan): BL
4) K-etchant GEL+G-Multi PRIMER (GC, Tokyo, Japan): GMP

After the application of surface treatments according to the manufacturers’ instructions (Table 1), twelve micro-bore Tygon tubes (Saint-Gobain Performance Plastics, Nagano, Japan) with a height of 0.5 mm and an internal diameter of 0.8 mm were set on each LDS surface to prepare the resin composite cylinder. A syringe-type resin composite (Clearfil AP-X, Kuraray Noritake Dental) was carefully filled into each tube with a resin-filling instrument (TMDU type3 #3, YDM, Tokyo, Japan) while ensuring that the resin composite was free of air bubbles using an 8× microscope (Inoue Attachment, Tokyo, Japan) and was then light-cured for 20 s. In this research, a light-emitting diode (LED) light-curing unit (Bluephase 20i, Ivoclar Vivadent) with high mode (1,200 mW/cm²) was used for light-curing. After light-curing, the Tygon tubes were carefully removed with a sharp blade. The LDS specimens were submerged in distilled water for 24 h at 37°C. Then, each group was subdivided into two subgroups (n=36): without thermocycling (immediate) and with thermocycling (5,000 cycles). The group subjected to thermocycling (immediate) and with thermocycling was free of air bubbles using an 8× microscope (Inoue Attachment, Tokyo, Japan) while ensuring that the resin composite was free of air bubbles using an 8× microscope (Inoue Attachment, Tokyo, Japan) and was then light-cured for 20 s. In this research, a light-emitting diode (LED) light-curing unit (Bluephase 20i, Ivoclar Vivadent) with high mode (1,200 mW/cm²) was used for light-curing. After light-curing, the Tygon tubes were carefully removed with a sharp blade. The LDS specimens were submerged in distilled water for 24 h at 37°C. Then, each group was subdivided into two subgroups (n=36): without thermocycling (immediate) and with thermocycling (5,000 cycles). The group subjected to thermocycling was placed in a thermal-cycling device (K178-08, Tokyo Giken, Tokyo, Japan) utilizing two water baths of 5°C and 55°C, respectively, with a dwell time of 30 s at each temperature prior to the micro-shear bond strength test.

For the micro-shear test, as previously described by Shimada et al.28), the specimens were attached to the universal testing machine (EZ-SX, Shimadzu, Kyoto, Japan) and then subjected to testing at a crosshead speed of 1 mm/min. When a specimen broke before the micro-shear bond strength test could be conducted, it was recorded as a pretesting failure (ptf), which was included as 0 MPa in the statistical calculations of the micro-shear bond strengths. The mean bond strengths and the survival percentage of each group were calculated.

Fracture mode analysis
After the testing procedures, the fractured-specimens were inspected under a scanning electron microscope (SEM; JSM-IT100, JEOL, Tokyo, Japan) to verify the failure mode. The mode of failure was classified as follows: adhesive (A), cohesive in bond (B), or cohesive in resin composite (R). ME and GMP did not have a bond layer. The area of each failure type (%) was calculated.

SEM observation of treated surface
To evaluate the micromorphological structure of LDS, twelve LDS specimens were treated with one of the four surface treatment agents as described above (three specimens per group), with the exception of light irradiation of UB. Afterward, the surface was ultrasonically rinsed with absolute acetone for 1 min to dissolve the adhesive for SEM observation. After gold-sputter coating, both back-scattered and second electron SEM images were obtained with a 20-kV voltage at 5,000× magnification to examine the features of the treated surfaces.

Surface roughness
For the evaluation of surface roughness, the surfaces of twenty-five LDS specimens were ground up to 0.3 µm with polishing papers infused with aluminum oxide grain (Lapping Film Sheet, 3M, St. Paul, MN, USA) under water rinsing. This was followed by ultrasonication in distilled water for 3 min and air drying. Five LDS specimens were left as control. Subsequently, twenty LDS specimens were treated with each surface treatment agent as described above (five specimens per group), except light irradiation of UB, and were ultrasonically rinsed with absolute acetone for 1 min. The quantification of the surface-roughness parameter Sa (arithmetic mean deviation) was done using a confocal laser scanning microscope (CLSM, VK-X150, Keyence, Osaka, Japan) equipped with a 20× objective lens. Three different areas of a specimen, the dimension of each area being 506 µm width×675 µm length, were scanned, and the Sa values were obtained using automatic multifile analysis software (VK-H1XM, ver.1.3.1.120, Keyence). The three Sa values per specimen were averaged, and five specimens for each group were used for statistical analysis.

Statistical analysis
Data were statistically analyzed in SPSS software (SPSS 22, IBM, Armonk, NY, USA) at α=0.05 significance level. For micro-shear bond strength test, a resin composite cylinder was considered as the statistical unit, and the data were analyzed using Dunn’s test with Bonferroni correction. Fisher’s exact test with Bonferroni correction was used to analyze the survival percentages of the micro-shear bond strength test specimens. Tukey’s test was employed to analyze Sa values.

RESULTS
Micro-shear bond strength test
The mean and standard deviation values of micro-shear bond strengths for all groups are presented in Table 2. The survival percentages of the specimens are presented in Table 3. ME and BL exhibited significantly higher initial bond strengths than those of UB and GMP (p<0.05). After thermocycling (5,000 cycles), the ME did not show a significant difference (p>0.05), but BL and GMP demonstrated a considerable decrease in bond strength (p<0.05). All specimens showed ptf in UB after 5,000 thermocycles. ME and BL showed significantly higher survival percentages than that of UB, and after 5,000 thermocycles, ME and BL showed significantly
higher values than that of GMP (p<0.05). The failure mode results are presented in Table 4. The predominant failure mode was an adhesive failure in all groups. The typical failure modes observed with SEM are shown in Fig. 1.

**Table 2** Means and standard deviations of micro-shear bond strength (MPa)

|        | TC0            | TC5000        |
|--------|----------------|---------------|
| ME     | 25.5±10.7*     | 24.0±7.3*     |
| UB     | 0.8±0.7        | N.A.          |
| BL     | 30.7±8.5**     | 17.9±7.5**    |
| GMP    | 13.3±9.9c      | 3.0±1.4d      |

Different uppercase superscript letters indicate significant differences among the rows (p<0.05). * indicates no statistical difference within each column (p>0.05).

N.A.: No data available due to debonding from the ceramic surface during thermocycling.

**Table 3** Number of specimens produced, number of specimens of pre-test failures and survival percentage

|        | TC0                        | TC5000                    |
|--------|----------------------------|----------------------------|
| ME     | 36                         | 36                        |
|        | Number of pre-test failures | 0                         | 0                          |
|        | Survival percentage        | 100%a                     | 100%c                      |
| UB     | 36                         | 36                        |
|        | Number of pre-test failures | 9                         | 36                         |
|        | Survival percentage        | 75.0%b                    | N.A.                       |
| BL     | 36                         | 36                        |
|        | Number of pre-test failures | 0                         | 36                         |
|        | Survival percentage        | 100%a                     | 100%c                      |
| GMP    | 36                         | 36                        |
|        | Number of pre-test failures | 6                         | 16                         |
|        | Survival percentage        | 83.3%A                    | 55.6%bd                    |

Different uppercase superscript letters indicate significant differences within the rows (p<0.05). Different lowercase superscript letters indicate significant differences within the columns (p<0.05). N.A.: No data available due to debonding from the ceramic surface during thermocycling.

**Table 4** Failure mode (area%)

|        | A       | B       | R       |
|--------|---------|---------|---------|
| TC0    |         |         |         |
| ME     | 79.4    | —       | 20.6    |
| UB     | 100     | 0       | 0       |
| BL     | 92.4    | 7.6     | 0       |
| GMP    | 91.7    | —       | 8.3     |
| TC5000 |         |         |         |
| ME     | 85.8    | —       | 14.2    |
| UB     | 100     | 0       | 0       |
| BL     | 92.5    | 4.2     | 3.3     |
| GMP    | 75.5    | —       | 24.5    |

A: adhesive failure, B: cohesive failure in bond, R: cohesive failure in resin composite

**Table 5** Average roughness (Sa) values

|        | ME       | UB       | BL       | GMP     | control |
|--------|----------|----------|----------|---------|---------|
| Sa (µm)| 0.17±0.031| 0.11±0.014 | 0.10±0.007 | 0.09±0.011 | 0.11±0.037 |

Same superscript letters indicate no significant differences (p>0.05).

**SEM observation of treated surface**

Representative SEM images with backscattered electron detector and secondary electron detector of four groups are shown in Figs. 2 and 3. All groups showed a surface with some scratches produced by grinding with 1000-grit silicon carbide paper. In
Fig. 1 Representative SEM images (100×) of typical failure modes. 
A: adhesive failure, B: cohesive failure in bond, R: cohesive failure in resin composite.

Fig. 2 SEM images with backscattered electron detector of LDS surfaces according to surface treatment (magnification: 5,000×). 
a: ME, b: UB, c: BL, d: GMP.

Fig. 3 SEM images with secondary electron detector of LDS surfaces according to surface treatment (magnification: 5,000×). 
a: ME, b: UB, c: BL, d: GMP.

Fig. 4 Representative CLSM 3D surface profiles of morphology after surface treatment. 
a: ME, b: UB, c: BL, d: GMP, e: No pretreatment.

addition, ME had microporosities and an incomplete crystal phase (Figs. 2a, 3a).

Surface roughness
The mean and standard deviations of Sa values are presented in Table 5. The surface profiles of 3D are shown in Fig. 4. ME showed significantly higher Sa values (Sa=0.17) than those of the others (p<0.05).

DISCUSSION
The application of a silane-coupling agent is known to improve the bond strength of LDS and silica-based ceramics^9,12,29^). It was reported that chemical bonds between LDS and a resin composite could be achieved by the silanol group of silane molecules reacts with silica on the ceramic surface to form siloxane bonds and these methacryloyl groups react with methoxy groups of resin^30^). Although all surface treatment agents used in the current study contained a silane-coupling agent, the
results were material dependent. ME and BL showed significantly higher initial bond strength than UB and GMP, and ME had higher durability than BL and GMP after 5,000 thermocycles. Thus, the null hypothesis of the current study was rejected.

ME combines ammonium polyfluoride and silane (trimethoxysilylpropyl methacrylate) in one bottle to simplify the process of etching for the glass-ceramics, hence resulting in fewer steps than those involved in the HF etching+silanization technique, which is the conventional method of surface treatment of LDS. Several studies have reported that ME showed topographic changes in SEM micrographs, which were less significant than those of HF since ME contains ≤10% of tetrabutyl-ammonium dihydrogen trifluoride, an acid used to obtain an irregular pattern on the ceramic surface and micromechanical retention. A study found that ME promoted the stability of resin bonding with LDS even after aging, which is consistent with our results. Moreover, our study presented that the mean Sa value of ME was higher than that of the other groups. It is suggested that ME exhibited long-term durability due to the micromechanical retention that was caused by the etching effect of ammonium polyfluoride. ME is partly composed of trimethoxysilylpropyl methacrylate that forms a thin silane layer to chemically bond with the ceramic after water rinsing and drying of the treated surface. The reaction mechanism of ME is not fully clear; however, a carbon residue was detected on the ME-treated ceramic surface by X-ray photoelectron spectroscopy, suggesting that trimethoxysilylpropyl methacrylate functioned effectively on the LDS surface.

UB showed significantly lower initial bond strength. All specimens showed ptf after 5,000 thermocycles. The UB material is a universal adhesive that includes 2-hydroxyethyl methacrylate (HEMA), 10-methacryloyloxydecyl dihydrogen phosphate (MDP), and γ-methacryloxypropyl trimethoxysilane (γ-MPTS) in addition to water in one bottle. However, it was reported that the shelf-life of the adhesive would have reduced if a silane-coupling agent, including γ-MPTS, was placed in the same bottle with an acidic solution, as a result of hydrolysis. Therefore, it would be ineffective in optimizing the bonding to silica-based ceramic materials such as LDS.

BL showed higher bond strength values than GMP. This can be explained by the reaction process of the silane-coupling agent in which the silanes first undergo hydrolysis followed by condensation to be able to perform as a coupling agent at the interface between inorganic and organic substances. As a silane-coupling agent, BL contains γ-methacryloxypropyl triethoxy silane (γ-MPTES) with an ethoxy group and GMP contains γ-MPTS with a methoxy group. The alkoxy groups of γ-MPTES or γ-MPTS are progressively hydrolyzed into silanol groups in case of acidic monomers presented in the same media. The subsequent silanol groups will form siloxane bonds with the hydroxyl groups of silicon oxide. BL consists of two bottles; one bottle contains an acidic monomer while the other includes silane-coupling agent and water. The silane-coupling agent in one bottle was activated by the acidic monomer in the other bottle. Alternatively, GMP contains both acidic monomer and γ-MPTS in the same bottle without water. The activation of γ-MPTS in GMP depends on the existence of water on the LDS surface. The silane-coupling agent and the acidic monomers must preferably be prepared in two separate bottles and then mixed just before their application to the ceramic surface. This may result in an efficient chemical-activation of the silane-coupling agent on the ceramic surface.

With regard to the limitations of this study, ME was the most effective ceramic surface treatment to LDS, however it cannot be used intraorally. In the situation of intra-oral repair of LDS, BL that contains γ-MPTES and acidic functional monomers in separate solutions was effective. In the future, the development of new materials that lead to micromechanical retention and intra-oral use is expected.

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