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Original

Enhancement of the Bonding Durability of Resin-based Cement to Lithium Disilicate Glass Ceramics Using Surface Modification Methods

Kotaro SATO, Akihiro FUJISHIMA, Yasuhiro HOTTA*
and Takashi MIYAZAKI

Abstract: The aim of the present study was undertaken to investigate the effects of several surface modification methods applied to lithium disilicate glass ceramics (LDCs) on their bonding durability with resin-based cement. The LDC specimens were sectioned using a low-speed diamond cutting machine, crystallized by heating in a furnace, and then subjected to several surface modification treatments such as acid etching, sandblasting, or silica coating with silane coupling agents. Then, a cylindrical titanium with a sandblasted surface was bonded to the surface-modified LDC specimens using resin-based cement. After being either stored in water at 37°C for 24 h or subjected to 10,000 and/or 30,000 thermal cycles, the specimens were subjected to a shear bond strength test using a universal testing machine. The surface-modified LDC specimens obtained by combining blasting and silica coating modification treatments showed excellent durability, similar to that of acid-etched specimens. For safer laboratory practice, we recommend using the silica coating method for LDCs prior to applying silane coupling agents.

Key words: lithium disilicate ceramics, surface modification, silane coupling agent, silica coating, blasting, acid etching

Introduction

In recent years, all-ceramic restorations have been popular worldwide with the introduction of new ceramic materials and new processing technologies, particularly the dental CAD/CAM systems. Fabricating all-ceramic restorations, while satisfying both esthetics and morphology requirements, is technically sensitive using conventional dental porcelains. Owing to recent developments in both the hardware and software of digitizers and CAD/CAM devices, all-ceramic restorations can now be routinely fabricated with satisfactory fit by using the newly available dental CAD/CAM systems¹⁻⁴.

Among the materials considered for processing by CAD/CAM systems, lithium disilicate glass ceramics (LDCs) seem promising because of their excellent esthetics⁵⁻⁷. Owing to the high bending strength of LDCs (i.e., 360-400 MPa), which is nearly three times that of conventional dental porcelain, LDCs can be employed for both single crown copings and posterior bridges using both CAD/CAM systems and the press systems⁸.

Department of Conservative Dentistry, Division of Biomaterials and Engineering, Showa University School of Dentistry, 1-5-8 Hatanodai, Shinagawa-ku, Tokyo 142-8555, Japan.

* To whom corresponding should be addressed.

Table 1. Materials tested in this study

Name	Code	Classification	Composition	Batch No,	Manufacturer
IPS e-max CAD	LDC	lithium disilicate ceramics	SiO ₂ , Li ₂ O, Al ₂ O ₃ , P ₂ O ₅	N57631 N18412 N36173	Ivoclar Vivadent
Epicord		Primer	MDP, acetone	0171BA	Kuraray Noritake
Espesil		Silane coupling agent	γ -MPS, ethanol	A418279	3M ESPE
Porcelain Etch		Etching	Hydrofluoric Acid	B6M4R	ULTRADENT
Linkmax		Resin cement	Composite	A1106011	GC
Rocatec pre		Alumina sand blast	110 μ m particle alumina sand	385205	3M ESPE
Rocatec plus		Tribochemical treatment	110 μ m particle silica coated alumina sand	229783	3M ESPE
Siranopen		Silicoater treatment	Flame spraying treatment with silica	63239	Bredent

As resin-based cement is mainly available for bonding LDC restorations to an abutment tooth⁹⁾, the bonding surface characteristics of LDC are important. Different surface treatments, involving primers and silane coupling agents, are available and reported to enhance the bonding strength of resin cement to porcelain and zirconia¹⁰⁻¹³⁾. These surface modification methods include acid etching, sandblasting, tribochemical treatment, and the application of phosphoric acid monomer¹⁴⁻¹⁷⁾. In particular, acid etching using hydrofluoric acid solution has been recommended to enhance the surface bonding characteristics of LDCs^{18, 19)}. However, because hydrofluoric acid is dangerous for humans, surface modification of restorative devices using hydrofluoric acid is not recommended for clinical practice if alternative methods are available.

This study aimed to evaluate the effect of several surface modification methods on increasing the bonding strength of LDCs to resin-based cement and to maintain bonding durability under procedural conditions.

Materials and methods

Specimen preparation

Table 1 lists the materials used in this study. LDC blocks (IPS. e.max CAD LT A3 C14, Ivoclar Vivadent, Schaan, Liechtenstein) were cut using a low-speed, diamond cutting machine (Isomet, Buehler, Chicago, IL, USA). The sections were then crystallized by heating in a furnace (Programat P300, Ivoclar Vivadent), followed by embedding in an acrylic tube adjusted for the LDC outer periphery and then filled with cold-curing resin (Palapress Vario, Heraeus Kulzer, Hanau, Germany). After polymerization, the specimens were cleaned ultrasonically in water for 10 min, and then allowed to dry naturally. Vinyl adhesive tapes (thickness: 70 μ m; hole diameter: 6 mm; Vinyl patches, Nichiban, Tokyo, Japan) were taped onto the specimens to define the

Table 2. Surface modification methods examined in this study

Code	Surface treatment	Surface modifications	Silane coupling agent	Primer
NON	None	None	None	None
SCA	None	None	Espesil	None
ASB	Rocatec pre	None	Espesil	None
HFT	None	Hydrofluoric Acid solution	Espesil	None
SLP	Rocatec pre	Silano pen	Espesil	None
TBL	Rocatec pre	Rocatec plus	Espesil	None
CBT	Rocatec pre	Rocatec plus	Espesil	Epricord

bonding area before surface modification.

Surface modification

Table 2 lists the surface modification methods examined in this study. Abbreviations employed for the specimens treated by different surface modification processes and associated details of the modification method are described as follows.

1. NON

NON specimens were prepared without any surface modifications and were used as the control samples.

2. SCA

SCA specimens were obtained by introducing a silane coupling agent (Espesil, 3M ESPE, Neuss, Germany) for 5 min onto the bonding surface, followed by drying with air blasting.

3. HFT

HFT specimens were obtained by treatment of the bonding surface with hydrofluoric acid solution (Porcelain Etch, Ultradent, South Jordan, UT, USA) for 1 min, followed by rinsing with water, drying, and application of a silane coupling agent (Espesil, 3M ESPE).

4. ASB

ASB specimens were obtained by sandblasting alumina powder, with a mean particle size of 110 μm (Rocatec pre, 3M ESPE), using an alumina sandblasting device (Rocatec Junior, 3M ESPE) at a pressure of 0.40 MPa (5 sec/cm²) and a distance of 10 mm, followed by application of a silane coupling agent (Espesil, 3M ESPE).

5. SLP

SLP specimens were prepared by subjecting the LDC specimen to a silica coating treatment, achieved by a special gas emission method using a Silano pen (Bredent, Senden, Germany). The special gas, containing minute silica particles, was emitted following alumina sandblasting (Rocatec pre, 3M ESPE) at a pressure of 0.40 MPa (5 sec/cm²) and a distance of 10 mm, and then the silane coupling agent (Espesil, 3M ESPE) was applied.

6. TBL

TBL specimens were obtained by conventional sandblasting using alumina powder with a mean particle size of 110 μm (Rocatec pre, 3M ESPE) and an alumina sandblasting device

(Rocatec Junior, 3M ESPE), followed by tribochemical treatment of the LDC specimen using silica-coated alumina particles with a mean size of 110 μm (Rocatec plus, 3M ESPE), at a pressure of 0.28 MPa (13 sec/cm²) and a distance of 10 mm. Treatment with the silane coupling agent (Espesil, 3M ESPE) followed.

7. CBT

CBT specimens were obtained using the same process as that employed for obtaining the TBL specimens, followed by drying with air blasting and application of phosphoric acid ester monomer (Epricord, Kuraray Noritake, Miyoshi, Aichi, Japan) to the bonding surface.

Preparation of bonding body

The bonding body material employed was JIS grade 2 cp titanium cylinder (KS-50, Kobelco, Japan) with a diameter of 8 mm and height of 2.5 mm. The surface of the bonding body was blasted with alumina powder with a mean particle size of 250 μm using a sandblasting device (Easy Blast, BEGO, Bremen, Germany) at a pressure of 0.40 MPa (5 sec/cm²) and distance of 10 mm. The bonding body was cleaned ultrasonically in acetone solution for 10 min, dried with air, and subsequently subjected to application of phosphoric acid ester monomer (Epricode, Kuraray Noritake) to the bonding surface.

Mounting

Mixed resin cement (Linkmax, GC, Japan) was applied to the LDC bonding surface in the area delineated by the vinyl adhesive tape, and then pressed into the titanium bonding body. The specimen was immediately loaded at 3 kgf using a constant loading device, and the excess cement was removed. Resin cement was irradiated from four directions for 20 sec each using a light-curing unit (Cure Master, Yoshida, Japan), for a total exposure time of 80 sec.

Shear bond strength test

The 144 specimens prepared were subjected to a shear bond strength test and placed in deionized water at 37°C for 24 h. Two other groups of specimens were subjected to a thermal cycle test (10,000 and 30,000 cycles), performed by immersing the specimens in deionized water at 5°C and 60°C for 1 min.

Fig. 1 and 2 schematically demonstrate the device used for the shear bond strength test, performed using a universal testing machine (1125-5500R, Instron, Japan) operating at a crosshead speed of 1.0 mm/min. The shear bond strength was measured by dividing the load at which failure occurred in the bonding area.

Statistical analysis

Statistical analysis was performed on the shear bond strength measured using Tukey's multiple comparison tests to determine the significant difference ($P < 0.05$) among data obtained for the different surface modification methods examined herein.

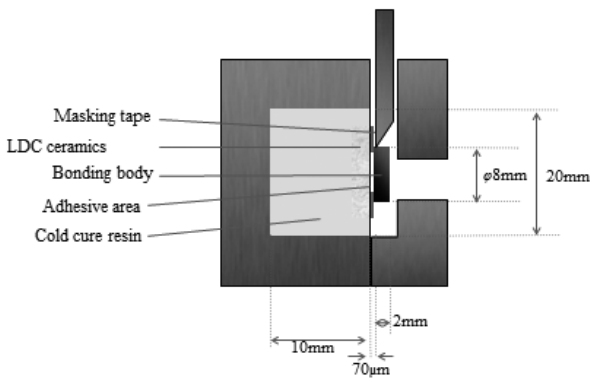


Fig. 1. Schematic of the shear bond-strength testing device

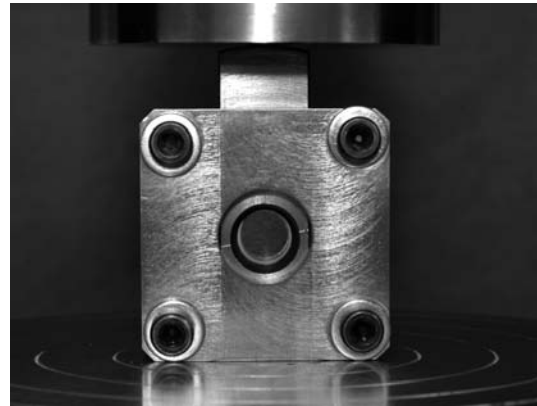


Fig. 2. Photographic image of the shear bond-strength testing device

Surface observation

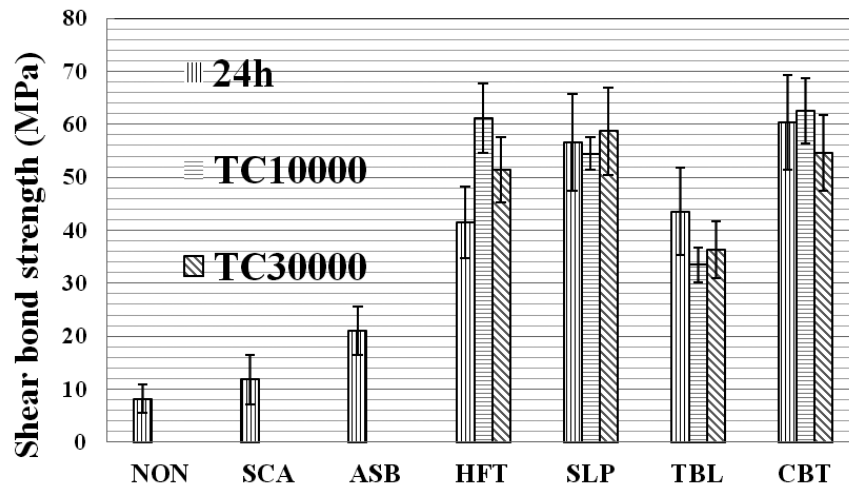
The mean surface roughness of the non-modified and modified LDC specimens was evaluated by a surface texture-measuring instrument (Surfcom, 480A, Japan). Backscattered electron images of the modified LDC surface were obtained using a scanning electron microscope (Miniscope[®] TM3000, HITACHI, Japan). Quantitative analysis of the modified LDC surface was performed using energy-dispersive X-ray spectroscopy (SwiftED3000, Oxford).

Results

Fig. 3 shows the shear bond strengths of the non-modified and modified LDC specimens. Following storage of the LDC specimens in water at 37°C for 24 h (24-h samples), the shear bond strengths of the modified samples were significantly higher than those of NON (control) specimens. The shear bond strengths of CBT and SLP were significantly higher than those of TBL and HFT, which were in turn significantly higher than those of ASB. There were no significant differences in the shear bond strengths between CBT and SLP or HFT and TBL.

After thermal cycling for 10,000 times (TC10000 samples), the NON, SCA, and ASB specimens showed debonding. The shear bond strengths of CBT, HFT, and SLP were not significantly different from each other, but were all significantly higher than that of TBL. Additionally, there were no significant differences in the shear bond strengths among SLP, TBL, and CBT. The shear bond strength of TC10000 HFT samples increased significantly compared with the corresponding 24-h HFT samples, whereas no significant effects on shear bond strength were measured in the TC10000 SLP, TBL, and CBT samples compared with the corresponding 24-h samples.

After thermal cycling for 30,000 times (TC30000 samples), the shear bond strengths of SLP, CBT, and HFT were again not significantly different from each other, but were all significantly higher than that of TBL. In addition, the shear bond strengths of HFT, SLP, TBL, and CBT TC30000 specimens were comparable with the corresponding 24-h samples, and those strengths except for HFT, were equivalent to the TC10000 specimens.



	NON	SCA	ASB	HFT	SLP	TBL	CBT
24h	8.2 (2.7) ^a	11.8 (4.7) ^a	21.1 (4.6) ^b	41.5 (6.8) ^c _A	56.5 (9.1) ^d _A	43.6 (8.3) ^c _A	60.3 (9.0) ^d _A
TC10000	Debonding	Debonding	Debonding	61.2 (6.6) ^e _B	54.5 (3.1) ^e _A	33.5 (3.2) ^f _A	62.6 (6.2) ^e _A
TC30000	Debonding	Debonding	Debonding	51.4 (6.1) ^e _{AB}	58.7 (8.3) ^e _A	36.4 (5.4) ^b _A	54.6 (7.1) ^e _A

Fig. 3. Shear bond strengths of the non-modified and modified LDC specimens. In the table supplied, across a particular row, samples denoted by the same superscript letters are not significantly different ($P > 0.05$). Across a particular column, samples denoted by the same subscript letters are not significantly different ($P > 0.05$).

Table 3. Mean surface roughness (R_a) of the bonding surface of the non-modified and modified LDC specimens. The samples denoted by the same superscript letters are not significantly different ($P > 0.05$).

Surface treatment	NON	ASB	SLP	HFT	TBL
Surface roughness (R_a ; μm)	0.14 (0.01)	2.86 (0.15) ^a	2.77 (0.11) ^{ab}	0.31 (0.01)	2.67 (0.15) ^b

Notably, the fracture surface of the cp titanium bonding body after the shear bond strength testing was completely covered with resin cement.

Table 3 shows the mean surface roughness of the non-modified and modified LDC specimens. The mean surface roughness of NON, HFT, ASB, SLP, and TBL specimens were 0.14 ± 0.03 , 0.31 ± 0.01 , 2.86 ± 0.15 , 2.77 ± 0.11 , and $2.67 \pm 0.15 \mu\text{m}$, respectively. There were no significant differences in the mean surface roughness values among ASB, SLP, and TBL.

Table 4 shows the results from the quantitative element analysis of the non-modified and modified LDC specimens. Relative to the NON specimens, the amount of Al was higher on the surface of ASB because of the alumina sandblasting method applied, while the Si content was higher on SLP and TBL surfaces relative to that on ASB because of the silica coating applied. In contrast, the Si content decreased and that of F increased on HFT surfaces, suggesting that

Table 4. Quantitative analysis of the bonding surface of the non-modified and modified LDC specimens

Mass concentration	Al	Si	P	K	F
Surface treatment					
NON	4.3	75.3	5	11	
ASB	9	73.4	5.1	8.4	
HFT	4.5	51.2	2.7	7.4	34.2
SLP	8.4	74.3	5	4	
TBL	8.2	76.6	4.4	7.4	

hydrofluoric acid solution selectively reacts with Si.

Fig. 4 shows the backscattered scanning electron microscopy (SEM) images of the non-modified and modified LDC surfaces. Fig. 4a shows the LDC surface before applying the silane coupling agent or phosphoric acid monomer. As observed in Fig. 4b, ASB featured many major cracks on the surface, and a rougher surface texture when compared with NON (Fig. 4a); although TBL and SLP (Fig. 4c, d) displayed similar features to those displayed by ASB, the cracks were smaller. In addition, HFT specimens (Fig. 4e) featured a characteristic spot-like micro crack morphology owing to the acid etching.

Discussion

All-ceramic restorations based on dental porcelain or porcelain layering on zirconia occasionally suffer from problems such as chipping and cracking because of the low mechanical properties of porcelain^{20,21}). In contrast, LDCs have excellent machinability, mechanical properties, optical properties, and chemical resistance upon heat treatment of lithium silicate glass ceramics²²).

With improvements in the mechanical and optical properties of LDCs now matching those of enamel, LDCs are used widely in the fabrication of prostheses with esthetic requirements. For instance, LDC crowns are fabricated by dental CAD/CAM systems. In addition, techniques for joining machined LDC veneering parts to machined zirconia frameworks using the CAD/CAM process were reported recently^{23,24}).

Hydrofluoric acid treatment is a popular method for the surface modification of LDCs worldwide. However, owing to safety concerns surrounding the handling of hydrofluoric acid in the laboratory and clinic, other surface modifications available for LDCs were herein evaluated as potential alternative treatments to the hydrofluoric acid treatment.

In this study, titanium was used as the bonding body for the shear bond strength testing and fracture mode observation due to titanium's demonstrated properties of strong adhesion to resin cement with primer composed with MDP²⁵). Self-adhesive types of resin-based cements, containing functional monomers, have become popular because of their ease of handling. However, in this study, resin-based cement with a relatively lower content of functional monomers was used to improve the bonding strength to ceramics²⁶).

Various treatment methods are available for effective adhesion of resin-based cement to dental

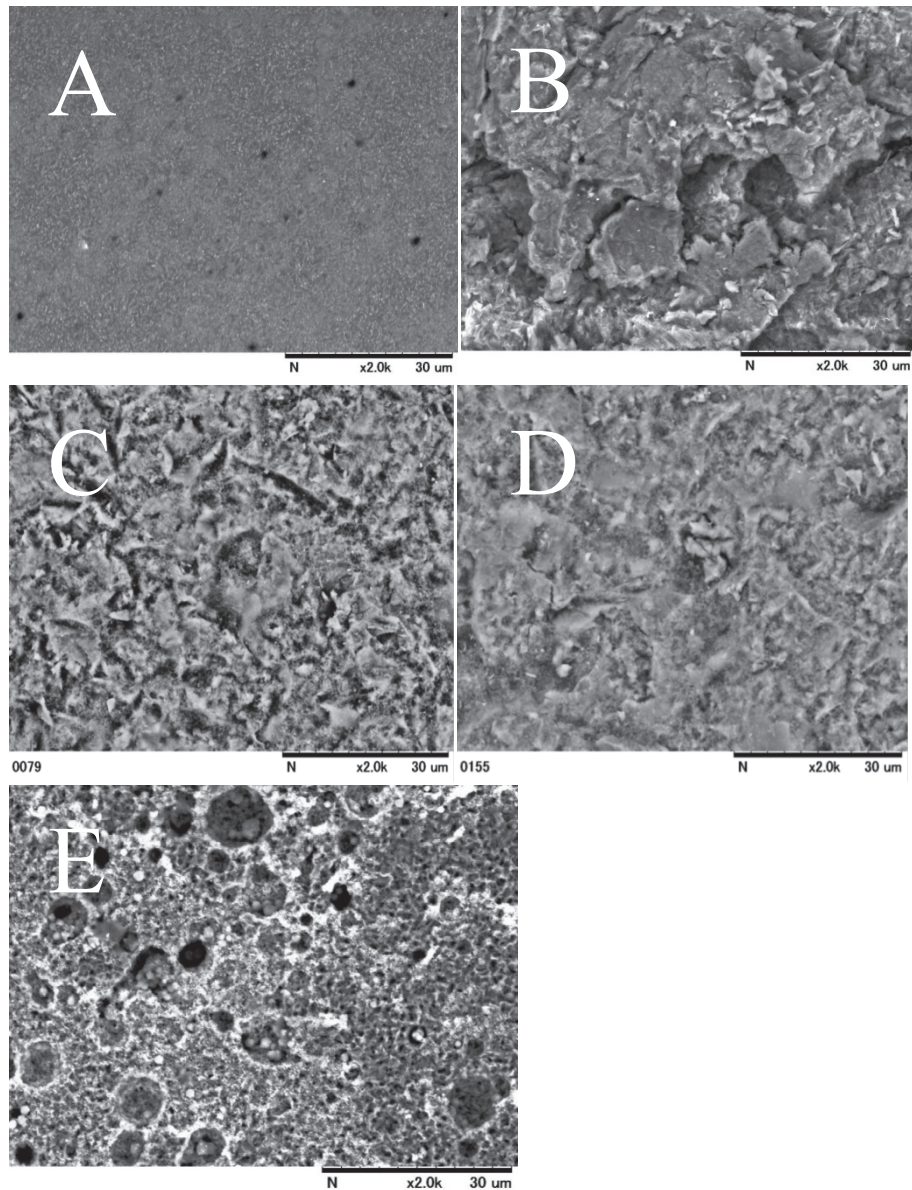


Fig. 4. Backscattered electron images of the non-modified and modified LDCs, (A) NON, (B) ASB, (C) TBL, (D) SLP, and (E) HFT. Magnification: 2000 \times .

materials. Alumina sandblasting is commonly applied to metal substrates because of the resulting surface area enhancement effects. Furthermore, silane coupling agents have been used as functional primers to chemically bond ceramics containing Si to resin-based cement.

As the shear bond strength of SCA was similar to that of NON following storage of the specimens in water at 37°C for 24 h, and SCA showed debonding after thermal cycling, we could deduce that application of the silane coupling agent only on the surface of LDC was not useful. Thus, an additional treatment was necessary to activate the silane coupling agent with a Si group

on the ceramic surface. This could be achieved using an acid environment, via dehydration condensation reactions²⁷⁾.

Modification of ceramic surfaces with Si molecular layers is known to enhance the effects exerted by the silane coupling agent^{17,21)}. To achieve a silica coating, various methods can be used, such as the Silano pen special gas emission method (used in this study) and the tetraethoxysilane flame spraying method. In addition to the silica coating treatment, this study used a tribochemical treatment that is essentially the same as the alumina sandblasting process, but using a silica-coated alumina powder in place of pure alumina powder. Similarly, the ceramic surface can be easily coated with silica using the same sandblasting device, operating at a slightly lower pressure.

The Rocatec system is commonly employed for applying a silane coupling agent to the formed layer using the tribochemical treatment²⁸⁾. Shimakura *et al*¹⁶⁾ and Takeuchi *et al*¹⁷⁾ reported that a combined treatment using MDP and silane coupling agent (Rocatec system) could enhance the bond strength and durability of yttria-partially stabilized tetragonal zirconia and silica-based glass ceramics. Therefore, such a combination treatment was used in this study.

Conventionally, increasing the surface area and applying a selected primer to the substrate surface are mandatory to achieve adhesion of resin-based cement. For dental metallic substrates, sandblasting is the most effective surface treatment for increasing the surface area for bonding to resin-based cement. However, sandblasting applied to brittle ceramics can be problematic, and sometimes results in material chipping. As shown in Fig. 3, the shear bond strength of ASB was significantly higher than that of SCA following storage of the specimens in water at 37°C for 24 h, although all samples suffered from debonding after thermal cycling. As shown in Fig. 4, the surface topography of ASB was rough, leading to increased surface area. Nevertheless, the application of silane coupling agent to the sandblasted surface was not effective. Therefore, further development of methods to effectively activate the silane coupling agent and improve adhesion to the substrate surface is needed.

According to Kumbuloglu *et al*¹³⁾, hydrofluoric acid is more effective than phosphoric acid for etching of LDCs, and is now the recommended treatment for increasing the surface areas of LDCs. As shown by SEM (Fig. 4), HFT featured a unique surface topography relative to the other specimens. Hydrofluoric acid reacts with the Si-based surface, resulting in an increased surface area and enhanced adhesive strength with micro retention effects²⁹⁾. Moreover, the shear bond strength of HFT was significantly higher than those of SCA and ASB, and was maintained even after thermal cycling. Phosphoric acid catalyst has been reported to enhance the effects of the silane coupling agent²⁷⁾. Our results now suggest that hydrofluoric acid solution also works as a catalyst for activating the silane coupling agent, thus generating excellent and durable adhesion.

Unfortunately however, hydrofluoric acid solution is biologically dangerous and can diminish the physical properties of the test specimen if the processing time is not carefully regulated³⁰⁾. Therefore, alternative methods for increasing the surface area and activating the silane coupling agent are still necessary.

The silica-based treatments studied herein, i.e., Silano pen and tribochemical treatments, enhanced the activity of the silane coupling agent. As observed in Fig. 3, the shear bond strengths of SLP and TBL were significantly higher than those of SCA and ASB, and were maintained after thermal cycling. Therefore, silica coating methods are useful to enhance the activity of silane coupling agents as primers.

While both silica coating methods tested herein were effective, the shear bond strength of TBL was significantly lower than that of SLP, and the repeated blasting processes may have damaged the surface structure and decreased the mechanical properties of TBL. For a deeper understanding of the process overall and how it could be made more effective, the effects of powder size and blasting pressure employed for the tribochemical treatment need to be investigated in the future.

Furthermore, the shear bond strength of CBT was significantly higher than that of TBL. The phosphoric acid ester monomer of CBT activated the silane coupling agent adsorbed onto the silica layer formed by the tribochemical treatment. Thus, the combined treatment had the same effects as hydrofluoric acid etching of LDC surfaces.

Conclusion

Surface modification methods applied to LDCs enhanced the bonding ability of LDCs to resin-based cement. In particular, silica coating methods using the special gas emission method and the tribochemical treatment combined with phosphoric acid ester monomer enhanced the activation effect of the silane coupling agent. The current study therefore demonstrates that applying alternative surface modification methods to hydrofluoric acid solution treatment can effectively modify the surface of LDCs to enable excellent adhesion properties to resin-based cements.

Conflict of interest disclosure

The authors declare no conflict of interest.

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