

Effects of Different Surface Treatments on Ceramic Repairs with Composite

Bi Rong HUANG¹, Xiao Yan WANG², Xue Jun GAO²

Objective: To compare different surface treatments of lithium disilicate-based ceramic on bond strengths to composite.

Methods: Lithium disilicate-based ceramic microbars (1 mm × 1 mm × 6 mm) were produced and abraded using diamond (D) or CoJet sandblasting (C), etched using phosphoric acid (P) or hydrofluoric acid (H) and silanised (S). A total of 12 surface treatments were adopted: D, DP, DH, DS, DPS, DHS, C, CP, CH, CS, CPS and CHS. An adhesive resin (XP Bond) was then applied and a composite build-up was placed. All specimens were thermocycled 5,000 times and submitted to microtensile bond strength test (μ TBS). Results were analysed using one-way analysis of variance and the Dunnett T3 test. Stereomicroscope and scanning electron microscopy were used to determine the failure modes.

Results: Surface roughening, acid etching and silanisation all have a significant influence on the ceramic-composite bond durability. Groups DH (30.7 ± 6.5 MPa), DHS (32.0 ± 8.2 MPa), CH (27.2 ± 4.5 MPa) and CHS (29.2 ± 5.5 MPa) showed the highest μ TBS statistically. Groups DS (11.5 ± 3.0 MPa) and DPS (9.7 ± 2.2 MPa) had higher μ TBS than groups D (4.5 ± 2.9 MPa) and DP (5.2 ± 3.0 MPa), while groups C (1.7 ± 0.7 MPa), CS (1.7 ± 0.9 MPa), CP (1.7 ± 0.6 MPa) and CPS (1.7 ± 1.1 MPa) showed the lowest μ TBS.

Conclusion: Hydrofluoric acid etching was effective in improving the bond strength of composite to lithium disilicate-based ceramic, regardless of the method of surface roughening and silanisation.

Key words: Ceramic repair, diamond, hydrofluoric acid, microtensile, silane

All-ceramic restorations have been popular in dentistry recently owing to their biocompatibility and aesthetic properties. However, all-ceramic restorations may still fail as a consequence of fractures, cracks, or chipping due to their brittle nature and structural flaws^{1,2}. Hot-pressed lithium disilicate-based ceramic (e.g. IPS

Empress 2 system), usually veneered with weaker porcelain is used as ceramic cores³. An *in vitro* study revealed that the lithium-disilicate-based bilayered crowns mostly failed between the veneer-core interface and the lithium disilicate-based ceramic core was exposed⁴.

The removal of fractured ceramic restorations may sacrifice the remaining sound tooth tissue and weaken the tooth. Repairing such restorations by bonding composites directly to the exposed ceramic is low cost, easy to perform and offers good aesthetics⁵. Therefore, while improvements to ceramics continue, it would be beneficial to have predictable means of repairing fractured ceramic restorations following the philosophy of conservative dentistry.

Recently, ceramic repair kits have been introduced in the dental market. These kits vary widely and various repair techniques have been recommended, including diamond surface roughening, hydrofluoric acid etching,

1 Department of Cariology and Endodontology, Peking University School and Hospital of Stomatology, Beijing, P.R. China; and Department of Preventive Dentistry, Beijing Stomatological Hospital, Capital Medical University, Beijing, P.R. China.

2 Department of Cariology and Endodontology, Peking University School and Hospital of Stomatology, Beijing, P.R. China.

Corresponding author: Dr Xiao Yan WANG, Department of Cariology and Endodontology, Peking University School and Hospital of Stomatology, #22 Zhongguancun Nandajie, Haidian District, Beijing 100081, P.R. China. Tel: 86-10-82195385; Fax: 86-10-62173402; E-mail: wangxiaoyan@pkuss.bjmu.edu.cn

phosphoric acid etching, silica coating and silanisation⁵. Several studies have proved hydrofluoric acid etching to be effective in improving the bonding between composites and glass-based ceramics⁶. When hydrofluoric acid is used intraorally, however, the patient and dentist may be at risk due to the toxicity and volatility of hydrofluoric acid⁷. Meanwhile, etching with 37% phosphoric acid along with the use of dental adhesives has been mostly used for tooth bonding and is not sufficient to condition the ceramic surface for reliable bonding⁸. Also, a silane coupling agent may provide sufficient immediate bond strength between composite and ceramic⁹. However, it may suffer hydrolytic degradation and its durability is therefore questionable¹⁰. So far, no optimal bonding protocol has been established for lithium disilicate-based ceramic repairs.

The microtensile bond strength (μ TBS) test has been widely used and is accepted as a predictable measuring tool of interfacial bond strengths¹¹. For ceramics, the cutting technique is time-consuming and may cause pre-test failures of specimens with low μ TBS¹². So, a non-cutting technique was used in this study for ceramic-composite specimen preparation for the μ TBS test. The effects of various ceramic-conditioning techniques on μ TBS of lithium disilicate-based ceramic to composite after thermal cycles were investigated. The null hypothesis was that ceramic surface treatment had no effect on ceramic-composite bond strengths.

Materials and methods

Ceramic-composite specimen preparation

A special ingot was designed to make the microbar index of wax with a central groove that was 1 mm in depth and 15 mm in length, and its width was regulated by screws (Fig 1). One hundred and eighty microbars (1 mm \times 1 mm \times 6 mm) of the lithium disilicate-based ceramic (IPS Empress 2, Ivoclar Vivadent) were fabricated in the EP 600 furnace (Ivoclar Vivadent) using the lost-wax technique. The ceramic microbars were air-abraded and ultrasonically cleaned to eliminate investment. All ceramic microbars were embedded in an impression material exposing the top surfaces, which were polished into 1,200 grit and were assigned to 12 groups (n = 15) as detailed in Table 1. The ceramic surfaces were abraded using a diamond (D) (TC11) or CoJet sandblasting (C) (3M EPSE), etched with 37% phosphoric acid (P) (Eco-Etch, Ivoclar Vivadent) or 9.6% hydrofluoric acid (H) (Porcelain Etch Gel, Pulpdent) and silanised (S) (Calibra silane, Dentsply). Then an adhesive resin

(XP Bond, Dentsply) was applied to the ceramic surfaces and light cured for 20 s at 700 mWcm⁻² (Translux CL, Kulzer). After the ceramic bars were carefully fixed into the groove of the ingot, the composite Ceram-X Mono (Dentsply) was inserted and light cured for 20 s at 700 mWcm⁻² (Fig 1c). All ceramic-composite bonded specimens were carefully picked out from the ingot and thermally cycled for 5,000 times between 5°C and 55°C with a dwelling time of 30 s.

Microtensile bond strength test

After thermal cycling, the bonding area of each specimen was measured using calipers before testing. The specimens were fixed on the flat plate of the microtensile tester (BISCO, USA) using cyanoacrylate adhesive (Super Glue, Taizhou Henco-glue). The ceramic-composite bonding interface was kept free and perpendicular to the force applied. The complex was loaded at a cross-head speed of 1 mm/min until fracture. Bond strength was expressed in MPa and calculated by dividing the load required for fracture the specimen (in N) by the bonded area (in mm²). The μ TBS values of specimens that failed prior to testing were recorded as zero.

The differences in μ TBS among the groups were statically analysed using one-way analysis of variance (ANOVA) and Dunnett T3 test (equal variances not assumed) at a significance level of 0.05 (SPSS 16.0).

Failure mode analysis

The fractured specimens were observed using a stereomicroscope (Zoom 630, Chang-Fang Optical) to analyse the failure modes. The failure modes were classified into adhesive failure (failure occurred within adhesive zone, between adhesive-ceramic interface and adhesive-composite interface), cohesive failure (failure occurred only in the ceramic or composite) and mixed failure (failure occurred crossing ceramic, adhesive and composite).

SEM observation

After the different treatments, the ceramic surfaces were observed by S-4800 model SEM (Hitachi). Also, typical images of fractured surfaces of ceramic-composite specimens were obtained by SEM. Samples were platinum sputter-coated prior to observation.

Results

The mean μ TBS and the failure modes for each group are shown in Table 2. In Group CPS, 2 of a total of 15 speci-

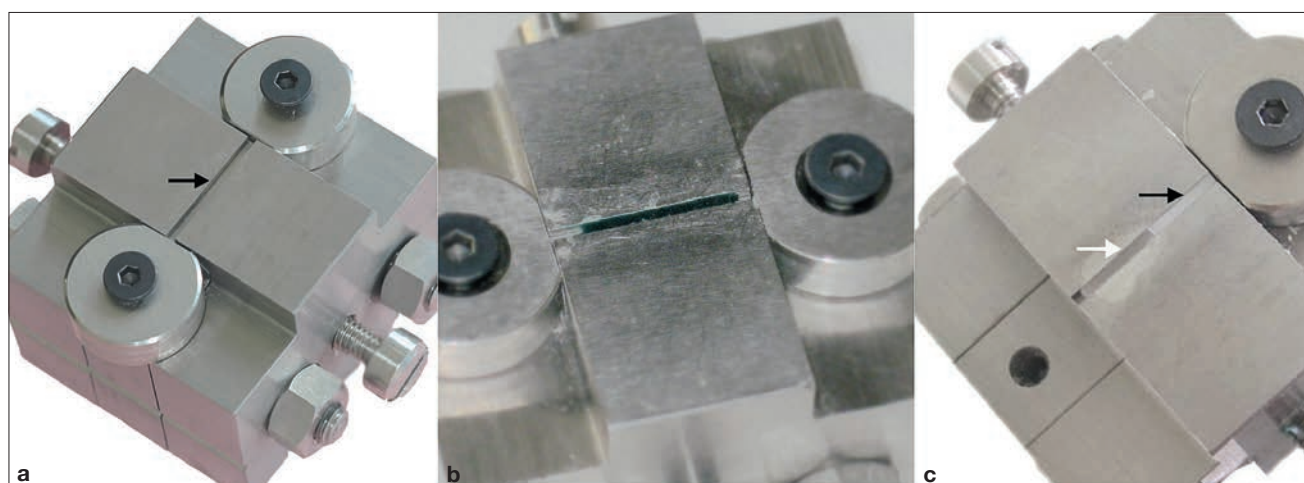


Fig 1 The specimen preparation for microtensile bond strength testing. **a** The central groove (black arrow) of ingot is used for preparing wax microbar and ceramic-composite bond specimen. **b** The production of the wax microbars. **c** The preparation of the microtensile test. The black arrow shows the ceramic, while the white arrow represents the composite.

Table 1 Different surface treatments of ceramic (experimental groups)

Group	Surface roughening	Acid etching	Silanisation
D	D	-	-
DP	D	P	-
DH	D	H	-
DS	D	-	S
DPS	D	P	S
DHS	D	H	S
C	C	-	-
CP	C	P	-
CH	C	H	-
CS	C	-	S
CPS	C	P	S
CHS	C	H	S

D: diamond abraded, ultrasonic cleaned with distilled water for 5 min and air dried.

C: coated by 30 μ m silica modified Al₂O₃ particles at a distant of less than 1 cm and a pressure of 100psi.

P: etched by 37% phosphoric acid for 60 s, water cleaned for above 20 s under high pressure and air dried.

H: etched by 9.6% hydrofluoric acid for 30 s, water cleaned for above 20 s under high pressure and air dried.

S: silanised the surfaces with Calibra silane for 60 s and air dried.

-: no treatment.

Table 2 Microtensile bond strengths and failure modes according to treatment groups

Group	n	μ TBS (MPa)	Failure modes		
			Adhesive	Cohesive	Mixed
D	15	4.5 ± 2.9 ^{a,d}	15	0	0
DP	15	5.2 ± 3.0 ^a	15	0	0
DH	15	30.7 ± 6.5 ^b	0	14	1
DS	15	11.5 ± 3.0 ^c	15	0	0
DPS	15	9.7 ± 2.2 ^c	15	0	0
DHS	15	32.0 ± 8.2 ^b	0	12	3
C	15	1.7 ± 0.7 ^d	15	0	0
CP	15	1.7 ± 0.6 ^d	15	0	0
CH	15	27.2 ± 4.5 ^b	1	10	4
CS	15	1.7 ± 0.9 ^d	15	0	0
CPS	15	1.7 ± 1.1 ^d	15	0	0
CHS	15	29.2 ± 5.5 ^b	0	13	2

Different superscript letters indicate statistically significant differences in μ TBS among groups (Dunnett T3 test, $\alpha = 0.05$).

* All the adhesive failures were ceramic-adhesive failure except for group CH. The cohesive failures were all within the composite.

mens debonded after thermocycling, and their μ TBS values were recorded as zero. No debonding of specimens was seen in the other groups. One-way ANOVA analysis revealed a significant influence of different surface treatments on the μ TBS: Groups DHS, DH, CHS, CH > Groups DS, DPS > Groups DP, D, and DP > Groups CS, C, CP, CPS ($P < 0.001$).

The majority of the failures in groups DH, DHS, CH and CHS were cohesive, while the remaining groups had a majority of adhesive failures. The cohesive failures were all within the composite. Adhesive failures were mostly between the ceramic and the adhesive except for 1 specimen from group CH. Typical SEM images of fractured surfaces of ceramic-composite specimens are shown in Fig 2.

SEM images of ceramic surfaces after treatment with diamond, CoJet, phosphoric acid and hydrofluoric acid etching are shown in Fig 3. The diamond abrasion resulted in a smear layer on the ceramic, which was not removed completely by phosphoric acid etching (Fig 3a and 3b). A granular layer on the ceramic surface was observed after CoJet sandblasting and phosphoric acid etching (Fig 3d and 3e). After hydrofluoric acid etching, the ceramics were eroded regardless of previous treatments (Figs 3c and 3f).

Discussion

In this study, different ceramic surface treatments did affect the ceramic-composite bond strength. The null hypothesis was therefore rejected.

Regarding the test performed, the stress distribution at the bonded interface in shear tests may be non-uniform, with the forces being in part cohesive directed at the base material rather than to the adhesive interface^{13,14}. These problems can be minimised with microtensile testing where testing of specimens with a bonded area of approximately 1 mm² results in more uniform stress distribution during loading¹⁵. However, it is hard to obtain specimens for microtensile test by the cutting method when bond strengths are lower than 5 MPa¹². A non-trimming method for preparing specimens is recommended to avoid any stress at the interface¹⁶. In the present study, a non-cutting method was used to prepare stick-shaped ceramic-composite specimens for microtensile test. The ceramic microbars were fabricated and bonded to the composite without cutting through the ceramic-composite interface. Results showed that only 2 of 180 specimens failed during thermocycling. The non-cutting preparation method should be considered for producing test specimens of which low bond strength values are expected. It is of particular interest in studies involving microtensile testing of ceramic specimens.

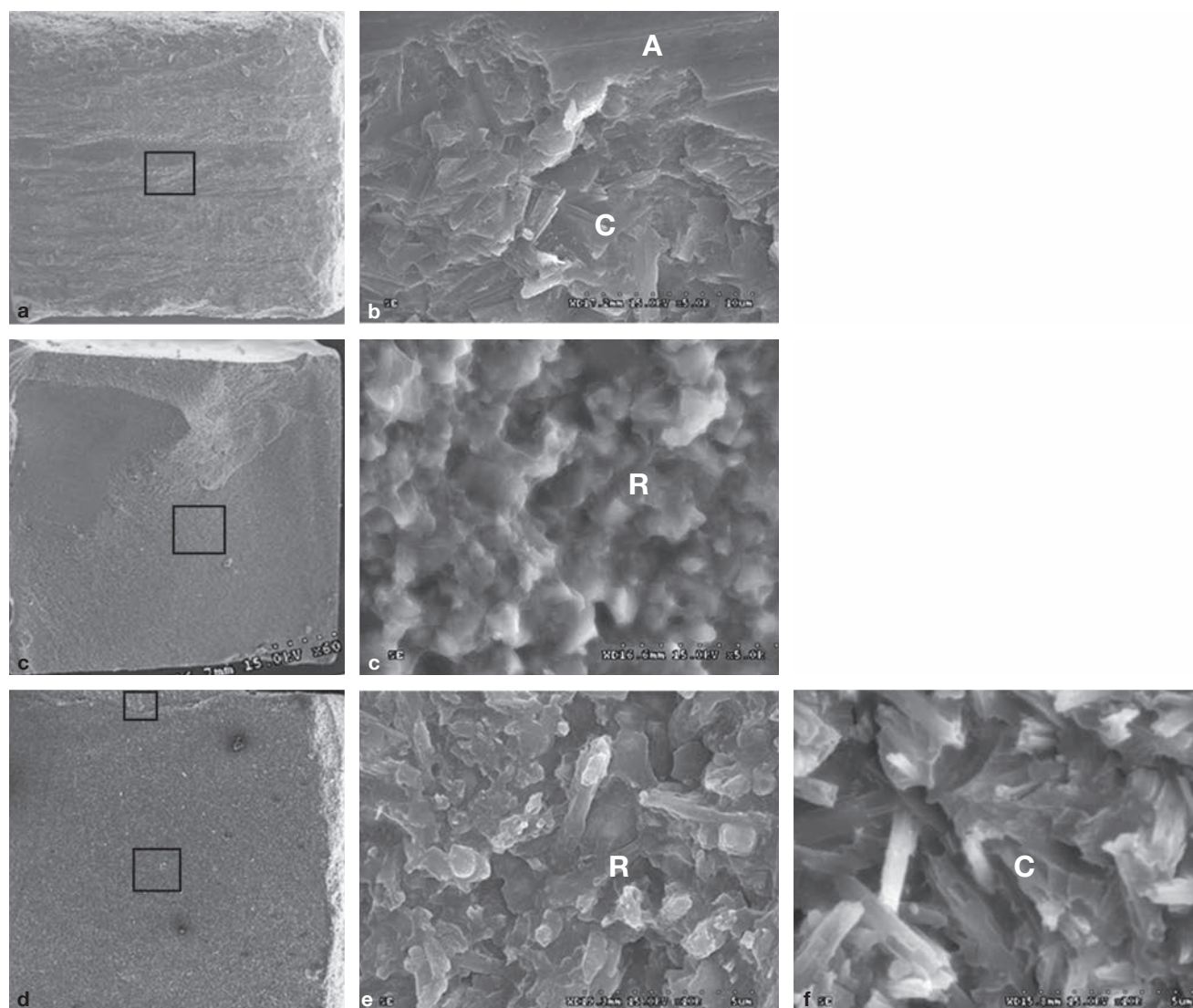


Fig 2 SEM observation of fracture surfaces. **a** Adhesive failure (70 \times). **b** Magnification of selected area of "a" (5,000 \times). **c** Cohesive failure (70 \times). **d** Magnification of selected area of "c" (5,000 \times). **e** Mixed-type failure (70 \times). **f** and **g** Magnification of selected area of "e" (10,000 \times). R: composite; A: adhesive; C: ceramic.

In this study, various ceramic surface treatments including roughening (diamond abrasion and CoJet sandblasting), acid etching (phosphoric acid, hydrofluoric acid and non-etching) and silanisation were used. Among the various treatments, DH, DHS, CH and CHS produced the highest bond strengths. This indicated that hydrofluoric acid etching was more effective than phosphoric acid treatment in improving the bond strength of composite to lithium disilicate-based ceramic, regardless of the method of surface roughening and silanisation^{17,18}. Using SEM, the cer-

amic surfaces etched by hydrofluoric acid were shown to be more irregular and significantly rougher than the samples etched by phosphoric acid after roughening with diamond or CoJet sandblasting. It has been reported that hydrofluoric acid can attack the glass phase of ceramics, partially dissolving it and creating microporous retention by exposing areas of crystals that make up the crystalline phase^{12,18}. The increased surface roughness is likely to have contributed to the micromechanical retention between the adhesive and the ceramic¹⁹.

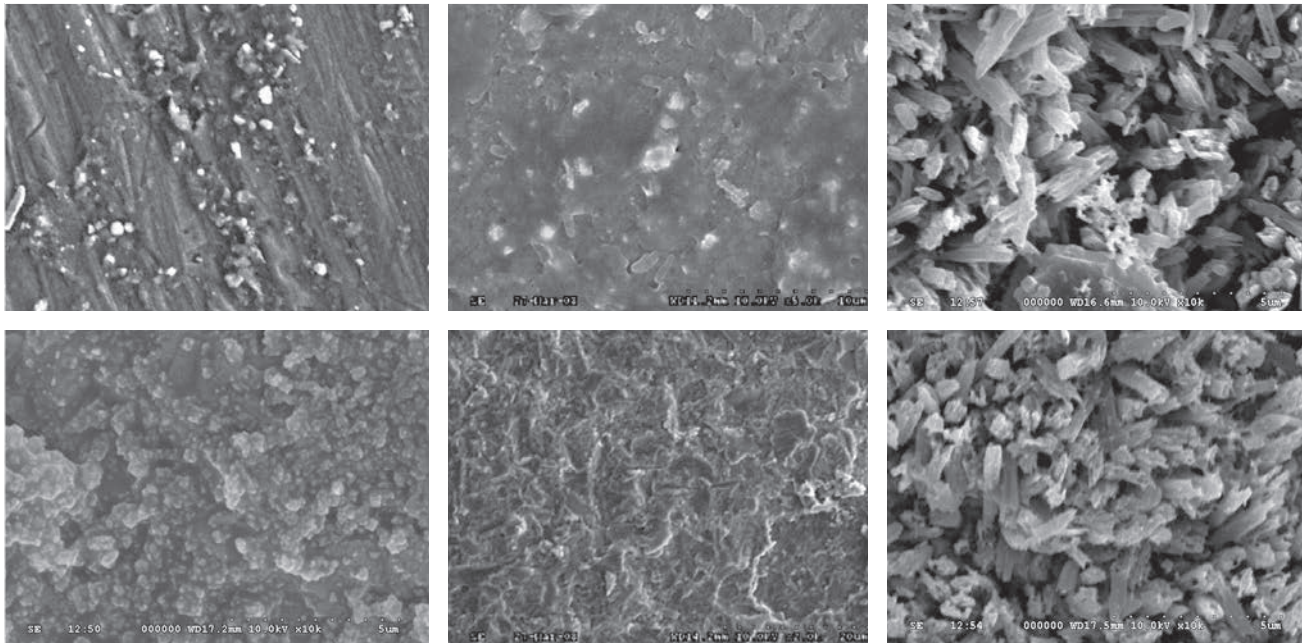


Fig 2 SEM images of ceramic surfaces after different treatments. **a** Diamond abraded (1,000 \times). **b** Diamond abraded followed by phosphoric acid etched for 60 s (5,000 \times). **c** Diamond abraded followed by hydrofluoric acid etched for 30 s (10,000 \times). **d** CoJet sandblasting (2,000 \times) **e** CoJet sandblasting followed by phosphoric acid etched for 60 s (2,000 \times). **f** CoJet sandblasting followed by hydrofluoric acid etched for 30 s (10,000 \times).

In contrast, phosphoric acid etching had little effect on enhancing the bond strength to lithium disilicate-based ceramic according to the present study. After etching with phosphoric acid, the ceramic surface was cleaned up but the surface roughness was not increased. It is known that there is no chemical reaction between phosphoric acid and ceramic material^{20,21}.

Tribochemical silica-coating techniques have been investigated for many years. CoJet and Rocatec are the systems mostly investigated and can produce a silica-coated ceramic surface with the high-speed injection of alumina-silica particles. Some studies indicated it could increase the bond strength between some ceramic materials (especially hydrofluoric acid-resistant ceramics) and resin luting agents^{22,23}. In the present study, the specimen treated with CoJet sandblasting, except for followed HF etching, showed low bond strength between the lithium disilicate-based ceramic and composite, which was also shown in a previous study (low bond strength values reported)²⁴. This indicated that the roughening effect of CoJet sandblasting on lithium disilicate-based ceramic may not be considered.

Silane coupling agents are adhesion promoters capable of forming chemical bonds with organic and inor-

ganic surfaces²⁵. Silanes can bond to composite by addition polymerisation reaction between methacrylate groups of the composite and the silane. Silane may also enhance ceramic-composite bonds by wetting the ceramic surface and thus facilitating the composite penetration into the microscopic pores in the ceramic²⁶. However, in this study the effect of silanisation on the ceramic-composite repair was dependent on the surface roughening and etching procedure used previously. The interaction of silane with ceramic that is treated differently should be further studied.

Within the limitations of this study, the following conclusions could be drawn: hydrofluoric acid etching was effective in improving the bond strength of composite to lithium disilicate-based ceramic, regardless of the method of surface roughening and silanisation. Compared with bur abrasion used solely, bur abrasion followed by silanisation could enhance the strength of the bond between lithium disilicate-based ceramic and composite resin. In practice, before applying the bonding agents on the lithium disilicate-based ceramic, etching by HF or a combined application of diamond abrasion and silanisation might be recommended for lithium disilicate-based ceramic repair.

Acknowledgements

The authors would like to thank Dr Dong PENG and Ms Qiao WANG from the Department of Prosthodontics and Dental Laboratory, Peking University School and Hospital of Stomatology for their assistance. The study was financially supported by the Dentsply Company.

References

- Harder S, Wolfart S, Eschbach S, et al. Eight-year outcome of posterior inlay-retained all-ceramic fixed dental prostheses. *J Dent* 2010;38:875–881.
- Beier US, Kapferer I, Dumfahrt H. Clinical long-term evaluation and failure characteristics of 1,335 all-ceramic restorations. *Int J Prosthodont* 2012;25:70–78.
- Heffernan MJ, Aquilino SA, Diaz-Arnold AM, et al. Relative translucency of six all-ceramic systems. Part I: core materials. *J Prosthet Dent* 2002;88:4–9.
- Zhao K, Pan Y, Guess PC, et al. Influence of veneer application on fracture behavior of lithium-disilicate-based ceramic crowns. *Dent Mater* 2012;28:653–660.
- Özcan M, Valandro LF, Amaral R, et al. Bond strength durability of a resin composite on a reinforced ceramic using various repair systems. *Dent Mater* 2009;25:1477–1483.
- Szep S, Gerhardt T, Gockel HW, et al. In vitro dentinal surface reaction of 9.5% buffered hydrofluoric acid in repair of ceramic restorations: a scanning electron microscopic investigation. *J Prosthet Dent* 2000;83:668–674.
- Brentel AS, Özcan M, Valandro LF, et al. Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions. *Dent Mater* 2007;23:1323–1331.
- Klosa K, Wolfart S, Lehmann F, et al. The effect of storage conditions, contamination modes and cleaning procedures on the resin bond strength to lithium disilicate ceramic. *J Adhes Dent* 2009;11:127–135.
- Yoshida K, Kamada K, Atsuta M. Effects of two silane coupling agents, a bonding agent, and thermal cycling on the bond strength of a CAD/CAM composite material cemented with two resin luting agents. *J Prosthet Dent* 2001;85:184–189.
- Matinlinna JP, Lassila LV, Özcan M, et al. An introduction to silanes and their clinical applications in dentistry. *Int J Prosthodont* 2004;17:155–164.
- Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength – evaluation of a microtensile bond test. *Dent Mater* 1994;10:236–240.
- El Zohairy AA, De Gee AJ, Mohsen MM, et al. Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks. *Dent Mater* 2003;19:575–583.
- DeHoff PH, Anusavice KJ, Wang Z. Three-dimensional finite element analysis of the shear bond test. *Dent Mater* 1995;11:126–131.
- Della Bona A, van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. *J Dent Res* 1995;74:1591–1596.
- Phrukkanon S, Burrow MF, Tyas MJ. The influence of cross-sectional shape and surface area on the microtensile bond test. *Dent Mater* 1998;14:212–221.
- Goracci C, Sadek FT, Monticelli F, et al. Influence of substrate, shape, and thickness on microtensile specimens' structural integrity and their measured bond strengths. *Dent Mater* 2004;20:643–654.
- Kim BK, Bae E, Shim JS, et al. The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials. *J Prosthet Dent* 2005;94:357–362.
- Panah FG, Rezaei SM, Ahmadian L. The influence of ceramic surface treatments on the micro-shear bond strength of composite resin to IPS Empress 2. *J Prosthodont* 2008;17:409–414.
- Ayad MF, Fahmy NZ, Rosenstiel SF. Effect of surface treatment on roughness and bond strength of a heat-pressed ceramic. *J Prosthet Dent* 2008;99:123–130.
- Kato H, Matsumura H, Atsuta M. Effect of etching and sandblasting on bond strength to sintered porcelain of unfilled resin. *J Oral Rehabil* 2000;27:103–110.
- Meng X, Yoshida K, Atsuta M. Microshear bond strength of resin bonding systems to machinable ceramic with different surface treatments. *J Adhes Dent* 2008;10:189–196.
- Özcan M, Alkumru HN, Gemalmaz D. The effect of surface treatments on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. *Int J Prosthodont* 2001;14:335–339.
- Rüttermann S, Fries L, Raab WH, et al. The effect of different bonding techniques on ceramic resin/shear bond strength. *J Adhes Dent* 2008;10:197–203.
- Blum IR, Nikolinakos N, Lynch CD, et al. An in vitro comparison of four intraoral ceramic repair systems. *J Dent* 2012;40:906–912.
- Jardel V, Degrange M, Picard B, et al. Correlation of topography to bond strength of etched ceramic. *Int J Prosthodont* 1999;12:59–64.
- Reston EG, Filho SC, Arossi G, et al. Repairing ceramic restorations: final solution or alternative procedure? *Oper Dent* 2008;33:461–466.