ELSEVIER

PII: S0300-5712(96)00078-4

Journal of Dentistry, Vol. 28, No. 1, pp. 53–58, 1998

Printed in Great Britain
0300–5712/98 $19.00+0.00

Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain

J.-H. Chen[†], H. Matsumura[‡] and M. Atsuta
Department of Fixed Prosthodontics, Nagasaki University School of Dentistry, Nagasaki, Japan

ABSTRACT

Objectives: The purpose of this study was to evaluate microstructure changes of Cerec 2 Vitablocs Mark II porcelain etched by a 5% hydrofluoric acid and examine the effect of different etching times on the bond strength between the porcelain and a composite resin.

Methods: Six different etching times (0, 5, 30, 60, 120 and 180 s) were used to etch the surfaces of the porcelain, respectively. Etched relief patterns were observed by means of a scanning electron microscope and the bond strength was determined between a dual-cured composite luting agent and the porcelain.

Results: The results showed that composite resin did not bond to unetched porcelain but bonded to etched porcelain, the 120 s etch giving the highest bond strength.

Conclusion: The bond strength values corresponded well to the microstructure changes caused by etching.

KEY WORDS: Adhesive, Bonding, Ceramics, Etching, Hydrofluoric acid, Porcelain

J. Dent. 1998; **26:** 53–58 (Accepted 21 October 1996)

INTRODUCTION

Both new porcelain restorative systems and adhesive ceramic restorations have greatly contributed to the increased interest in esthetic dentistry. Clinical success with these restorations, such as inlays, onlays, and laminate veneers, etc. has been assisted by the ability to develop a reliable bond of composite resin to the etched porcelain surface.

Although grinding and air abrasion[1–3], combined or not with acid etching, have been used on the bonding surface of a ceramic restoration to create a mechanically retentive surface for porcelain bonding[4–11], and silanes have also been added to enhance the bond strength chemically[1,12–18], many researchers consider etching the bonding surface as the critical treatment for bonding to composite resin[9–11]. In addition, a number of studies have led to observations that the bond strength of composite resin to etched porcelain exceeds the cohesive strength of porcelain[4,5,8,11–13]. Simonsen and Calamia[4,12], Calamia *et al.*[6], and Lacy *et al.*[7] established that although other acids could be used as feldspathic porcelain etchants, the best result could be obtained with the shortest etch time using hydrofluoric acid. In their studies, Simonsen and Calamia[4], and Calamia *et al.*[6] concluded that the etchant concentrations and etching times used for different porcelains were critical in obtaining the maximum bond strengths.

Unfortunately, in most of the current porcelain bonding research, the porcelains were etched with

Supported in part by a grant from the Japan–China Medical Association (#1851-1996).
[†]*Present address:* Prosthetic Department, College of Dentistry, The Fourth Military Medical University, People's Republic of China.
[‡]*Correspondence should be addressed to:* Dr H. Matsumura, Department of Fixed Prosthodontics, Nagasaki University School of Dentistry, 1-7-1, Sakamoto, Nagasaki 852, Japan. Tel.: +81-958-49-7688. Fax: +81-958-49-7689.

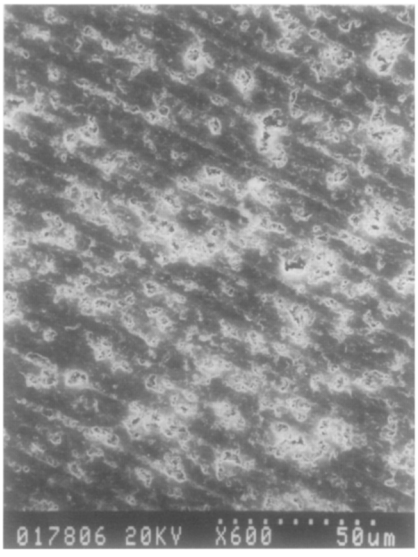

Fig. 1. A scanning electron micrograph of the unetched surface of the Cerec 2 Vitablocs Mark II porcelain.

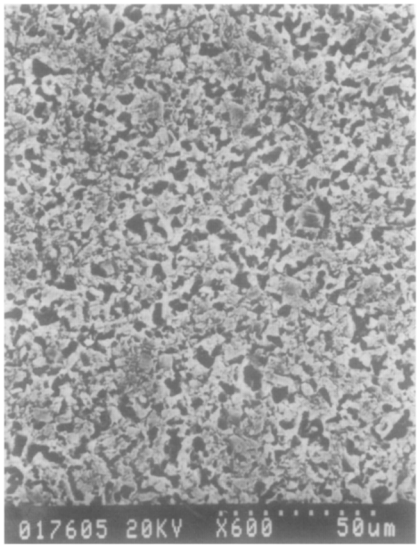

Fig. 3. After a 30 s etching, most of the areas are etched but many crystal areas still remain unetched.

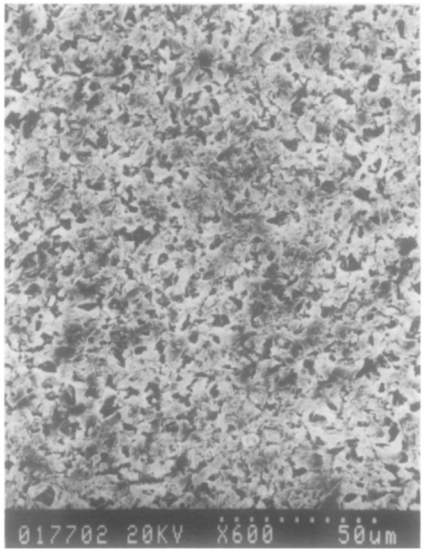

Fig. 2. Porcelain surface etched with 5% hydrofluoric acid gel for 5 s. Some cavities caused by etching can be clearly seen.

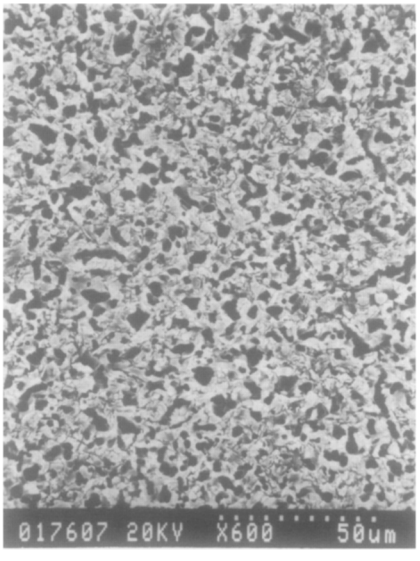

Fig. 4. After a 60 s etching, there are more etched cavities and undercuts than that shown in *Fig. 3.*

hydrofluoric acid for a time period prescribed by the manufacturer or decided by the researchers with no experimental basis available except for a few scanning electron micrographs (SEM)[7,11,13].

The purpose of this study was to evaluate the effect of different etching times on the microstructural changes of a machinable porcelain and on the bond strength between the porcelain and a resin composite with SEM and shear bond testing.

MATERIAL AND METHODS

The porcelain used in the present study was Cerec 2 Vitablocs Mark II (A3C I10 and A3C I8, Vita Zahnfabrik GmbH, Bad Säckingen, Germany), which was designed for the Cerec 2 system (Siemens AG, München, Germany). A 5% hydrofluoric acid gel (HF Gel, GC Corp., Tokyo, Japan) was used for etching the porcelain. The resin cement used was Clapearl DC (Kuraray Co. Ltd, Osaka, Japan), which was a two-paste dual-cured composite luting agent designed for seating ceramic restorations. Despite the recommendations of the manufacturer, the proprietary silane coupling agent was not used since the effect of porcelain surface acid etching on the bond between the composite resin and the porcelain was of interest.

Fifty-four rectangles were cut from the Vitablocs A3C I10 bars with a low-speed saw (Buehler Ltd, Lake Bluff, USA), each with a length of 10 mm by 8 mm in width and 2.5 mm in thickness. In addition, 48 square specimens ($8 \times 8 \times 2.5$ mm) were cut from the A3C I8 Vitablocs bars and ground with a dental stone into round shapes of 2.5 mm in thickness and 8 mm in diameter. The specimens were then divided into six

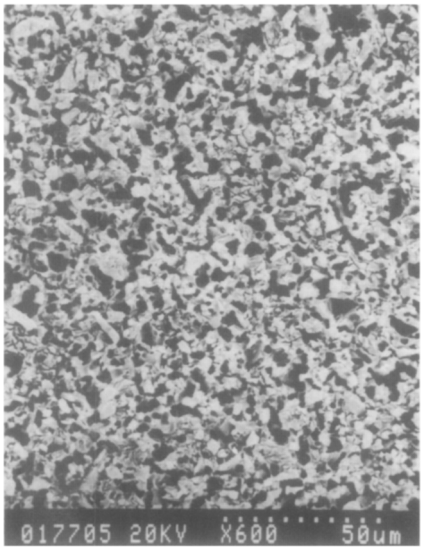

Fig. 5. After a 120 s etching, the generated cavities are distributed evenly in the crystals, therefore indicating a well etched surface.

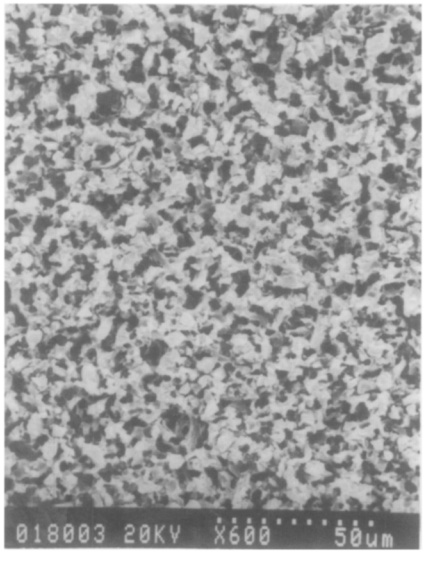

Fig. 6. After a 180 s etching, the porcelain surface is overetched and the crystals it contains are not as clear as those shown in *Fig. 5*.

groups of nine rectangular and eight round units each. After being ground flat with No. 600 silicon-carbide abrasive paper, the surfaces to be bonded were etched with 5% hydrofluoric acid for 0, 5, 30, 60, 120 and 180 s for groups 1 through 6, respectively. The etched specimens were washed with tap water for 1 min, cleaned by ultrasonic bath immersion in distilled water for 5 min, and dried with compressed air for 30 s.

One rectangular specimen was taken from each group, dried and sputter-coated with gold to be observed with an electron microscope (S-520, Hitachi Ltd, Tokyo, Japan) operated at 20 kV. Therefore, there were 16 specimens left in each group, with eight rectangular and eight round. A piece of tape with a circular hole 5 mm in diameter was fixed on the centre of each rectangular porcelain specimen to define the bond area and a consistent 50 μm thickness of luting material.

The luting cement was mixed as recommended by the manufacturer. A thin layer of luting cement was used to cover the hole area of the tape on the rectangular specimen as well as the surface of the round specimen. The two porcelain specimens were then pressed together with a 5 N load. After excess material was wiped away with a brush, they were photo-polymerized from two opposite directions each for 40 s using a visible-light source (Quick Light, J. Morita Corp., Osaka, Japan). The light fibre was fixed at a height of 10 mm to standardize the distance from its end to the bond interface.

The completed samples were stored in water at 37°C for 24 h before shear testing was undertaken. The shear bond strengths were then measured with a universal testing machine (DCS-500, Shimadzu Corp., Kyoto, Japan) at a cross-head speed of 0.5 mm/min by use of a shear testing jig (Wago Industrial Ltd, Nagasaki, Japan)[8]. The calculated shear bond strength was determined by dividing the maximal force applied during the shear testing by the bonding area. For each condition, the mean and standard deviation (SD) of eight tests were calculated. The bond strengths were then compared by one-way analysis of variance (ANOVA). When the *F*-test was significant, Duncan new multiple range intervals were further performed with the value of statistical significance set at $P=0.05$.

The fracture sites for all the specimens were observed with an optical microscope (SMZ 100, Nikon Corp., Tokyo, Japan) and were recorded with their corresponding fracture force. Fracture modes were categorized into five groups: (1) composite resin–porcelain interface separation; (2) fractured specimen, previously wrapped by self-cured acrylic resin for the purpose of fixation, the porcelain rectangles broke down into three parts, with the middle part still bonding to the round porcelain part; (3) the fractures occurred when the wrapped parts broke down into two parts with the bonded area remaining unaffected; (4) separation of porcelain–composite resin–porcelain interfaces with crack propagation inside the porcelains; (5) the fractures occurred as the round porcelain parts broke down.

RESULTS

Figs. 1–6 illustrate the porcelain surfaces etched for different times. *Fig. 7* shows the means and standard deviations for bond strengths in each group. Five kinds of fracture modes were recorded and summarized in *Fig. 8*. The shear bond strength corresponding to each fracture was also listed in *Table 1*.

The means and standard deviations of the shear bond strengths for groups 1–6 were given in *Table 2*. One-way ANOVA revealed that the bond strength was

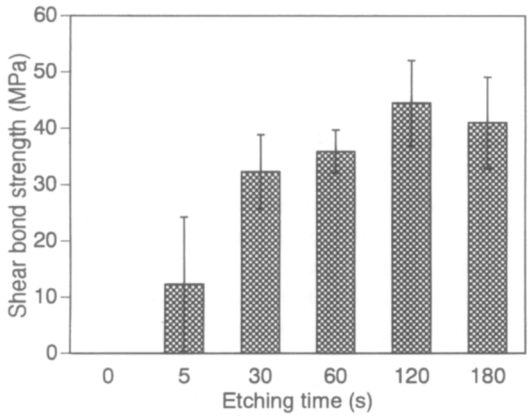

Fig. 7. Mean bond strengths of composite resin to the porcelain subjected to different etching times. Bars indicate the standard deviations.

significantly affected by the etching period (F=45.8, P=0.0001). It appears that the values having the same letters for Duncan grouping in the same column are not significantly different. All the bonded specimens in the no-etching group separated after they had been stored in water at 37°C for 24 h. Thus, shear bond strength was recorded for this group as 0 MPa. The values for group 2 were also very low, with a mean of 12.3 MPa. The highest mean shear bond strength was achieved when the porcelain surface was etched for 120 s.

Although almost all the fractures occurred in the porcelain of groups 4, 5 and 6, the mean of group 5 is still significantly higher than other groups except group 6. The means in groups 1 and 2 were significantly lower than that of all other groups (P<0.05).

DISCUSSION

The effect of different etching times on the shear bond strength between the Cerec 2 Vitablocs porcelain and composite resin is significant as shown by this study. The photomicrographs also clearly reveal the effect of different etching times on the microstructures of the porcelain.

As described by Yen *et al.*[19], the effect of etching can be explained by the chemical nature of the etching process. Hydrofluoric acid can react preferentially with the silica phase in feldspathic porcelain to form hexafluorosilicates. As a result, the surface of the porcelain becomes honeycomb-like, which is expected for microretentions. Compared with the micrograph of the air-abraded porcelain surface appearing in the Kato *et al.*[3] article, we found that the surfaces of porcelain etched for 120–180 s in the present study were rougher and the microundercuts appeared deeper.

Stangel *et al.*[13] also observed the microstructures of another feldspathic porcelain etched by different acids and for different times. Though the surfaces of the porcelain had been air-abraded for 20 s before acid etching, the changes in the microstructures of the porcelain etched for 120 and 180 s in the present study were very similar to those of the air-abraded samples which were etched for similar time periods by Stangel *et al.*[13]. This suggests that air-abrading of the porcelain surface may not be necessary for the purpose of bonding porcelain with luting cement if acid etching is used.

Such a hypothesis is supported by the fact that the shear bond strengths between porcelain etched by 5% hydrofluoric acid for 120–180 s and composite resin were superior to the strength of the adhered porcelain. Kern and Thompson[20] found that the abrasion rate of feldspathic materials by sandblasting was 36 times higher than that of In-Ceram ceramic. This observation led them to the conclusion that sandblasting of feldspathic materials should be avoided for the reason of volume loss and changes in morphology. In other words, etching the bonding surface of a restoration produced by feldspathic porcelain might be the only choice available to obtain micromechanical retention.

In the present study, it was found that within the range of 0–120 s, the longer the etching time, the higher the bond strength. At 180 s, however, etching resulted in a decrease of bond strength, though it was not significantly different from that of the 120 s etching group.

All the fractures recorded in groups 4–6 occurred in the porcelains, as shown in *Table 1*, except for one

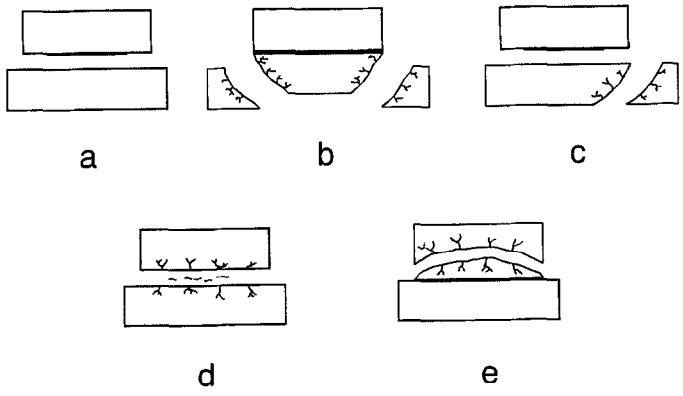

Fig. 8. Classification of bond fracture modes.

Table I. Distribution of fracture modes and the corresponding shear bond strengths

Fracture mode	*Group*	*Etching time (s)*	*Bond strength (MPa)*	*Number*
a	1	0	0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0	8
	2	5	3.9 4.0 5.5 6.0 7.3 10.2 24.5	7
	3	30	21.8 25.9	2
	4	60	32.0	1
b	2	5	36.7	1
	3	30	29.7 31.2 33.2 35.2	4
	4	60	33.8 35.5 35.7	3
	5	120	34.9 39.2	2
	6	180	32.5 42.7 45.2	3
c	3	30	40.6	1
	4	60	32.5 34.0 41.2 42.4	4
	5	120	39.7 44.0 45.9 59.2	4
	6	180	34.2	1
d	3	30	40.7	1
	5	120	42.5 51.0	2
	6	180	41.9 45.5	2
e	6	180	30.7 54.9	2

Each mode corresponds to the bond fracture mode sketched in *Fig. 8.*

Table II. Mean bond strengths (MPa) and statistical analysis results

Group	*Etching time (s)*	*Sample size*	*Mean*	*SD*	*Duncan grouping*
1	0	8	0	0	
2	5	8	12.3	11.9	
3	30	8	32.3	6.6	A
4	60	8	35.9	3.8	A B
5	120	8	44.5	7.6	C
6	180	8	41.0	8.1	B C

Duncan grouping: identical letters in the same column indicate that the values are not statistically different at P=0.05.

specimen in group 4. The fracture modes correspond well to the shear bond strengths. It is easy to understand that the shear bond strength may be lower when debonding occurs at the interface (*Fig. 8*a) due to a weak link between the composite and porcelain. However, it seems difficult to explain why debonding occurred more frequently by breakdown of a portion of the bonded specimens (*Fig. 8*b,c) rather than crack propagation inside the porcelains (*Fig. 8*d) and to break down another portion of the bonded specimens (*Fig. 8*c), since the shear bond strength recorded for these fractures are similar. As we found no records of such a relation between a fracture site and the shear bond strength, we regard that this irregularity is derived from variableness of a fracture starting point.

CONCLUSIONS

In this study, we observed the changes in the microstructure of Cerec 2 Vitablocs Mark II porcelain etched by a 5% hydrofluoric acid for different times and measured the shear bond strength between a composite resin and porcelain previously etched. The following conclusions were drawn from the experiment:

(1) The etched relief pattern observed through a scanning electron microscope indicated an increase in surface roughness with extension of etching time.
(2) The Clapearl DC composite luting agent did not bond to the unetched Cerec 2 Vitablocs Mark II porcelain. For the porcelain, the etching of the bonding surface was critical to bond strength.
(3) Bond strengths greater than 30 MPa were generated when the porcelain was etched with 5% hydrofluoric acid for more than 30 s.

References

1. Bertolotti, R. L., Lacy, A. M. and Watanabe, L. G., Adhesive monomers for porcelain repair. *International Journal of Prosthodontics*, 1989, **2**, 483–489.
2. Appeldoorn, R. E., Wilwerding, T. M. and Barkmeier, W. W., Bond strength of composite resin to porcelain with newer generation porcelain repair systems. *Journal of Prosthetic Dentistry*, 1993, **70**, 6–11.
3. Kato, H., Matsumura, H., Tanaka, T. and Atsuta, M., Bond strength and durability of porcelain bonding systems. *Journal of Prosthetic Dentistry*, 1996, **75**, 163–168.
4. Simonsen, R. J. and Calamia, J. R., Tensile bond strength of etched porcelain. *Journal of Dental Research*, 1983, **62** (Abstr. 1154), 297.

5. Ferrando, J. M. P., Graser, G. N., Tallents, R. H. and Jarvis, R. H., Tensile strength and microleakage of porcelain repair materials. *Journal of Prosthetic Dentistry*, 1983, **50**, 44–50.
6. Calamia, J. R., Vaidyanathan, J., Vaidyanathan, T. K. and Hirsch, S. M., Shear bond strength of etched porcelains. *Journal of Dental Research*, 1985, **64** (Abstr. 1096), 296.
7. Lacy, A. M., LaLuz, J., Watanabe, L. G. and Dellinges, M., Effect of porcelain surface treatment on the bond to composite. *Journal of Prosthetic Dentistry*, 1988, **60**, 288–291.
8. Matsumura, H., Kawahara, M., Tanaka, T. and Atsuta, M., A new porcelain repair system with a silane coupler, ferric chloride, and adhesive opaque resin. *Journal of Dental Research*, 1989, **68**, 813–818.
9. Sorensen, J. A., Engelman, M. J., Torres, T. J. and Avera, S. P., Shear bond strength of composite resin to porcelain. *International Journal of Prosthodontics*, 1991, **4**, 17–23.
10. Thurmond, J. W., Barkmeier, W. W. and Wilwerding, T. M., Effect of porcelain surface treatments on bond strengths of composite resin bonded to porcelain. *Journal of Prosthetic Dentistry*, 1994, **72**, 355–359.
11. Roulet, J. F., Söderholm, K. J. M. and Longmate, J., Effects of treatment and storage conditions on ceramic/composite bond strength. *Journal of Dental Research*, 1995, **74**, 381–387.
12. Calamia, J. R., Simonsen, R. J., Effect of coupling agents on bond strength of etched porcelain. *Journal of Dental Research*, 1984, **63** (Abstr. 79), 179.
13. Stangel, I., Nathanon, D. and Hsu, C. S., Shear strength of composite bond to etched porcelain. *Journal of Dental Research*, 1987, **66**, 1460–1465.
14. Diaz-Arnold, A. M., Schneider, R. L. and Aqullino, S. A., Bond strength of intraoral porcelain repair materials. *Journal of Prosthetic Dentistry*, 1989, **61**, 305–309.
15. Bailey, J. H., Porcelain-to-composite bond strengths using four organosilane materials. *Journal of Prosthetic Dentistry*, 1989, **61**, 174–177.
16. Suliman, A. H. A., Swift, E. J. Jr. and Perdigao, J., Effects of surface treatment and bonding agents on bond strength of composite resin to porcelain. *Journal of Prosthetic Dentistry*, 1993, **70**, 118–120.
17. Stacey, G. D., A shear stress analysis of the bonding of porcelain veneers to enamel. *Journal of Prosthetic Dentistry*, 1993, **70**, 395–402.
18. Aida, M., Hayakawa, T. and Mizukawa, K., Adhesion of composite to porcelain with various surface conditions. *Journal of Prosthetic Dentistry*, 1995, **73**, 464–470.
19. Yen, T. Y., Blackman, R. B. and Baez, R. J., Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. *Journal of Prosthetic Dentistry*, 1993, **70**, 224–233.
20. Kern, M. and Thompson, V. P., Sandblasting and silica coating of a glass-infiltrated alumina ceramic: volume loss, morphology, and changes in the surface composition. *Journal of Prosthetic Dentistry*, 1994, **71**, 453–461.