ORIGINAL ARTICLE

EFFECT OF THE PORCELAIN FITTING SURFACE ON THE PORCELAIN TO COMPOSITE BOND STRENGTH[1]

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إن القدرةفي الحصول على التصاق جيـــد بيـــن الحشـــوات أو الوجـــوه الخزفيـــة والكمبوزيت يساعد على استعمال هذه التعويضات في مجـــال الترميمـــات الســـنية المعتمدة على الالتصاق ٠

عدة طرق لمعالجة سطح التصاق الخزف بما في ذلك تخشين الســـطح أو معالجتـــه بالحمض أو تطبيق السلاين تم إظهارزيادة قوة التصاق الخزف بالكمبوزيت ٠

الخزف المخبوز على رقائق البلاتين تنتج سطحا أملس نسبيا أو علـــــى المســـحوق الكاسي فإنها تعطى سطحا خشنا٠

هذه الدراسة تختبر أهمية خشونة السطح المخبوز على قوة الألتصاق التـــي يمكـــن التوصل اليها بين سطح الالتصاق الخزف والكمبوزيت كمادة لاصقة وهي تقـــارن أيضا تأثير الطرق المختلفة لمعالجة سطح الالتصاق والمستعملة بشكل كبير لتحسين الألتصاق بين الخزف والكمبوزيت ٠

التفاصيل عن المواد المستخدمة في هذه الدراسة تم تلخيصها في جدول (١) ٠

مائة وستون صفيحة من الخزف بمقاس ٨ ملم × ٤ملم تم عملها حســـب الطريقـــة السابقة الذكر

تم تقسيم الصفائح بعد ذلك إلى مجموعتين رئيسيتين من سطح أملس إبتدائى (A) وسطح خشن إبتدائى (B) ٠وكل مجموعة رئيسية تحتوي على ثمانى مجموعـــات أخرى أعتماداً على طريقة السطح كما في جدول (٢) ٠ بعـــد ذلـــك تـــم وضـــع الصفائح في هذه المجموعات ٠

الأسطح المعالجة بطريقة الرمل المضغوط أو التخريش بحمض الـــهيدروفلوريك أو المعالجة بالسيلايين كل على حدة أو باستخدام أكثر من طريقة معاً ٠

وبعد معالجة السطح تم اختبارقوة الالتصاق ٠

وبشكل عام تظهر الأسطح الابتدائية إرتباطاً أضعف من الأسطح المعالجة ٠

بمقارنة مجموعات معالجة بنفس الطريقة من المجموعتين A ،B تري النتائج أن الأسطح الخشنة الأبتدائية ليست أقوى من الأسطح الملساء بالرغم من الأرقام العالية للأسطح الحشنة ٠

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Introduction

The ability to achieve a reliable bond between porcelain [ceramic] restorations and composite resins promoted the use of the latter to cement the former. Various ceramic fitting surface

Ceramic inlays and veneers made on platinum foil produce a smooth fitting surface finish while those made on refractory investment die give a rough fitting surface finish. These ceramic restorations are then bonded to teeth with composite resin cement. This study examined i) the influence of the fitting surface finish on the ceramic/composite resin interfacial bond; and ii) the effect on the bond strength achieved following various ceramic surface treatment methods such as gritblasting, hydrofluoric acid etching and silane priming either as single treatment or in various combinations. Shear bond strength test was used to assess the ceramic/composite interfacial resin bond. The refractory investment die [rough] surface group had higher bond strength than the smooth [polished] surface group although the difference was not statistically significant. The control for the smooth-fitting surface surface group had a mean bond strength of 0.23 megapascals which was lower than that of the control for the rough-fitting surface group which had a mean of 5.23 megapascals. When the polished fitting surface was treated with a combination of gritblasting, hydrofluoric acid etching and silane priming, the mean bond strength was 10.72 megapascals whereas the mean bond strength for the refractory investment die surfaces with a similar treatment was 15.96 megapascals. The bond strength achieved by the above combination was significantly higher than the respective control at $p<0.0001$. The following bond failures were found: a)adhesive at the ceramic/composite resin interface, b)cohesive within the ceramic and c)a combination of both. Cohesive bond failure within the ceramic made it difficult to differentiate the effect of the different types of surface treatment on the interfacial bond strength.

treatments including roughening, acid etching and the application of silanes had been shown to enhance porcelain to composite bond strength.[1-10] Ceramic veneers and inlays are either fired on platinum foil, which produces a relatively smooth surface finish, or on refractory investment dies which give a rough surface finish.[11-13] This study examined the significance of the surface topography and chemistry on the bond strengths achieved between the ceramic fitting surface and composite resin cement. It also compared the effect of different fitting surface treatments commonly used to improve the porcelain/ composite interfacial bond strength.

Materials and Methods

The details of the materials used in this study are summarized in Table 1. One hundred and sixty porcelain discs measuring 8mm x 4mm were fabricated according to a previously described method.[13] Group A was made up of 80 discs which surfaces were polished to represent the platinum foil or smooth fitting surface finish. Group B consisted also of 80 discs with a refractory investment or rough fitting surface finish. These groups were subdivided into eight comparable subgroups based on the type of surface treatment given. The treatments given to the ceramic bonding surfaces of the various subgroups are shown in Table 2. Treatments were gritblasting, hydrofluoric acid (HF) etching and silane priming either as a single treatment or in various combinations. Fitting surfaces of one subgroup of each group served as control for the respective group.

Mounting the disc in perspex

Each disc was mounted in perspex* with the aid of a plastic mold. The disc was placed in the center at the base of the mold. The liquid and transparent perspex material were poured slowly into the mold and monitored visually so as not to displace the ceramic disc. Refractory bonding surfaces were protected by attaching two cardboard pieces to their surfaces with a water soluble gum[1] during perspex mounting. They were later assigned to their respective subgroup and the pieces of cardboard were dissolved away with water after the perspex had solidified.

Disc surface preparation and treatment

Mimicking a platinum foil smooth fitting surface finish was achieved by grinding the mounted disc surface flat with silicon carbide paper of grades 400 and 600. Final polish was done with 1 um diamond compound* on the Metaserv Grinder-Polisher* at 100 revolutions per minute for not less than 30 minutes. Metadi fluid* was used as lubricant during polishing. Gritblasting of the bonding surfaces was achieved using 50 um aluminum oxide" at a pressure of 40 pounds per square inch.

Hydrofluoric acid (10%) was used to etch the specimens for 9 minutes as recommended by the manufacturer. After gritblasting and/or acid etching, the disc surfaces were washed under running laboratory-grade distilled water and then ultrasonically cleaned for 30 minutes using distilled water.For silane treatment, 37% phosphoric acid was applied to the dry surface of the disc. The acid was left on the disc surface for 90 seconds before adding the silane from the Porcelain Repair Primer kit. Both solutions were allowed to react for 60 seconds before washing off with distilled water and drying

'Polymethylmethacrylate, ICI, UK
tPelifix, Pelican AG, Hanover-1, West Germany
[J]Buehler-met AG, CH-4023, Basel, Switzerland
[§]Buehler, Conventry, England
"Korox, 50 Bego, Bremen, Germany

Table 1. Materials used.

Product	Batch No.	Manufacturer
Flexoceram investment material	1536	Elephant Ceramics, Hoorn, Holland
Flexoceram Inlay Porcelain		Elephant Ceramics, Hoorn, Holland
Bondlite unfilled Resin		Kerr Manufacturing Company, Michigan, USA
Porcelite Dual Cure		Kerr Manufacturing Company, Michigan, USA
Porcelain Repair Primer	14066	Kerr Manufacturing Company, Michigan, USA
Acid Etchant	81354	Kerr Manufacturing Company, Michigan, USA
Hydrofluoric Acid	2057232	Chemistry Department, University of Sheffield, England

Table 2. Grouping and sub-grouping of the samples.

Group A Polished Starting Surface	Group B Refractory Starting Surface
polished surface only- no treatment	untreated refractory surface
gritblasted surface only	gritblasted surface only
etched surface only	etched surface only
polished surface + silane priming	refractory surface + silane priming
gritblasted + etched surface	refractory surface + etched surface
gritblasted+ silane application	gritblasted + silane application
etched surface + silane priming	etched surface + silane priming
gritblasted + etched + silane treatment	gritblasted + etched + silane treatment

with air.

Bonding procedure

A 4 mm thick circular silicone tablet was made from heavy body silicone impression material.[1] Using a specially designed casting ring, a hole 3mm in diameter, was made in the center of the silicone tablet. The hole facilitated the application of the bonding composite resin to the ceramic surface. After the relevant disc surface treatment, the silicone tablet was placed on the ceramic disc and the perspex block and then the total assembly was transferred to a specially designed cementation alignment clamp. The perspex block was locked-in from beneath to keep it steady in the top part of the cementation alignment clamp. The cementation alignment clamp design also allowed composite resin to be added directly to the center of the ceramic disc through the silicone tablet which maintained the specific disc surface area required for the composite resin bonding as shown in Figures 1 and 2.

A drop of Bondlite unfilled resin and activator were mixed for 10 seconds. The mixture was added to the ceramic surface with a bristle brush through the hole in the cementation alignment clamps. The mixture was photo-polymerized for not less than 60 seconds with Visilux-2 light curing unit*. The base shade and catalyst of Porcelite dual cure cement were then mixed and applied in increments to the bonding surface until the resin reached the brim of the casting ring surface. Each increment was photo-polymerized for about 60 seconds.

Following bonding, the clamp was released and the bonded specimen carefully removed from the cementation alignment clamp as well as from thesilicone tablet with the aid of an amalgam

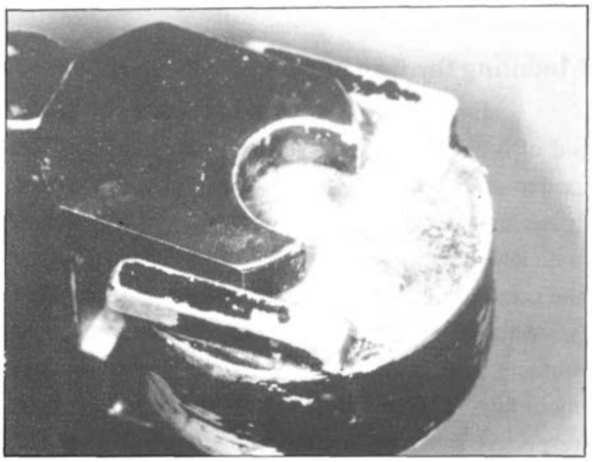

Figure 1. The cementation alignment clamp showing the 3mm hole through the silicone tablet for introducing composite resin to the ceramic base.

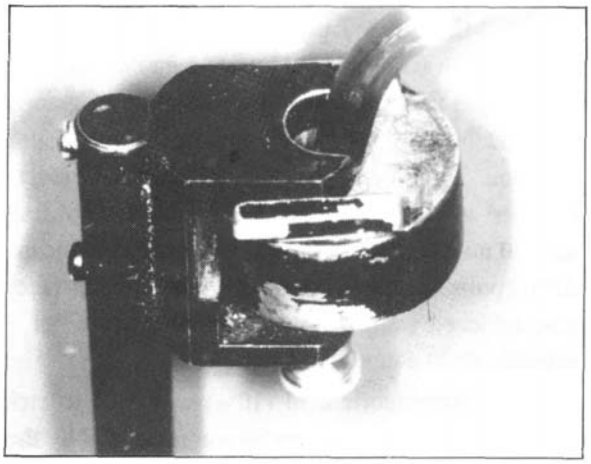

Figure 2. Photo-polymerization through the hole to the ceramic surface. The resin block is clamped from under its surface.

condenser applied on the perspex block to stabilize the composite resin portion of the specimen. Each specimen [Fig. 3] was allowed to stand on the bench for one hour and stored in a water bath at 37+l°C for at least 24 hours. The specimens were then thermocycled 500 times between 6 and 60° with a 30 second dwell time in each bath and 20 seconds travel time between baths prior to shear bond strength testing.

[1]Provil, Bayer, Berkshire, United Kingdom
*Dental Products Division, 3M, Minnesota, USA

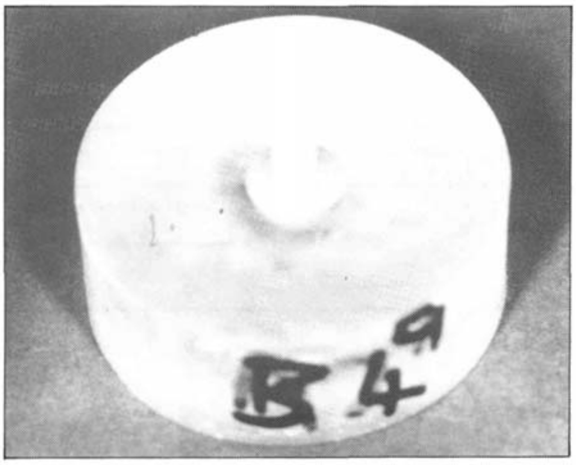

Figure 3. The resulting test specimen.

Bond strength testing

The specimens were transferred from the water bath to the holding jig of a Universal testing machine* after drying water from the surface. A shear load was applied at ceramic/composite resin bond interface parallel to the ceramic surface using the metal tip attached to the fixed arm of the testing machine [Fig. 4]. The machine operating in the shear compression mode was programmed for a cross-head speed of 1 mm/min. The maximum load at failure was recorded for each specimen.

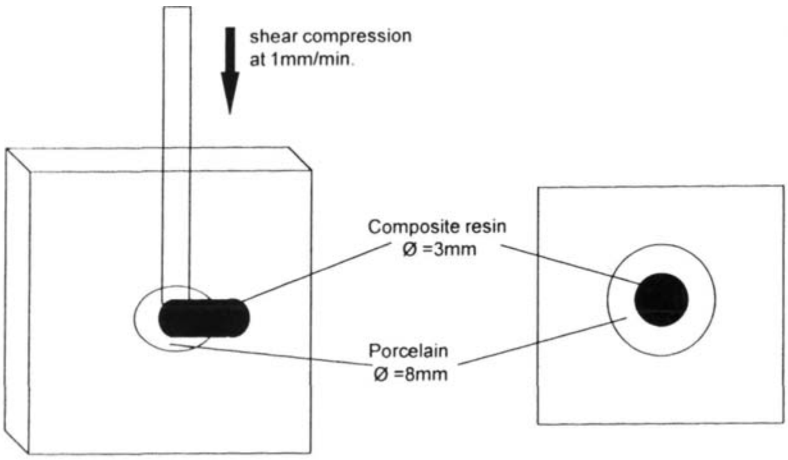

Figure 4. Schematic diagram of the test arrangement.

The failure loads were divided by the surface area of composite disc to determine the shear bond strength. The results were calculated in Megapascals (MPa) and analyzed for statistical significance of variations in the bond strengths achieved. Kruskal-Wallis one-way analysis for non parametric tests and the Scheffe multiple range test in the SPSS statistical software package* were used.

The pattern of bond failure was also determined with the aid of the optical microscope and recorded. The failure modes were categorized as adhesive [A] when failure occurred at the ceramic/composite resin interface; cohesive [C], when failure occurred solely within the ceramic; and mixed [M], that is adhesive and cohesive failures, when failure occurred with some retention of ceramic at the ceramic/composite resin bond interface.

Results

The results of the bond strength achieved are summarized in Tables 3-5. The bond strengths of group A specimens varied from 0.23 to 10.72 MPa. Highest strength value was recorded in the subgroup receiving combined surface treatment of

§Model 9M5K, J.J. Lloyd Instruments Ltd., Warsash, England

*SPSS Inc_ Cnlcaa_o_ "-• • USA

gritblasting, HF acid etching and silane priming. Group B specimens failed at strengths ranging from 5.23 to 15.96 MPa, with highest strength observed in the subgroup with a combined surface treatment of gritblasting and HF acid etching.

Subgroups had statistically significantly higher bond strength than their control surfaces except the subgroups that received only gritblasting. The bond strengths of acid-etched and silane-treated subgroups were significantly higher than their respective control surfaces at $p<0.05$. Subgroups treated with only silane showed bond strength that was significantly higher than the control surfaces at $p<0.001$.

When similarly treated subgroups from groups A and B were compared, the refractory surface subgroups were not stronger than the respective polished surface subgroups despite the higher bond strength figures of the refractory surface subgroups (Table 5).

The details of the modes of bond failure for the different subgroups are presented in Tables 3-5. Cohesive failure always occurred within porcelain. Mixed failures involved failure at the interface and cohesively within the porcelain. It was observed that as the shear bond strength increased, more cohesive fractures of the ceramic base were noticed. The etched-only surfaces did not fall within this trend as most specimens failed cohesively despite intermediate bond strength values (Tables 3 and 4).

Table 3. Porcelain to composite shear bond strength [MPa] Group A: (polished starting surfaces) (n=10)

Group	Mean	**+Std. Error**	Mode of Bond **Failure**		
			C	M	A
Polished	0.2276	+0.0560[3]	0	0	10
GBL	4.1144	+0.5358[a]	0	1	9
Etched	6.2862	+0.5990[b]	10	0	0
GBL/Etch	7.8602	+0.8399[b]	5	5	0
Etch/Silane	8.3172	+0.6203[b]	8	2	0
GBL/Silane	8.8294	+0.6622[b]	5	5	0
Silane	9.3733	+ 1.4499[b]	9	1	0
GBL/Etch/ Silane	10.7171	+1.3883[b]	10	0	0

Different alphabets show statistically significant differences

Table 4. Porcelain to composite shear bond strength [MPa] Group B: (refractory starting surfaces) (n=10).

Group	**Mean**	**+Std. Error**	**Mode of Bond Failure**		
			C	**M**	**A**
Refractory	5.2313	+ 1.2290[6]	**0**	0	10
GBL	10.2895	+0.8425[a]	0	**4**	6
Etched	11.8174	+ 1.1281"	8	2	0
GBL/Etch	11.7542	+0.9040[b]	5	5	0
Etch/Silane	13.7004	+ 1.1962[b]	10	0	0
GBL/Silane	14.7576	+ 1.3748[b]	10	0	0
Silane	14.5971	+1.1952[b]	9	**1**	0
GBL/Etch/ Silane	15.9556	+0.9629[b]	10	0	0

Different alphabets show statistically significant

Discussion

The results showed that the control surfaces were significantly weaker than the treated surfaces when they were subjected to shear bond strength tests. Previous studies[1"1014] had shown that various ceramic fitting surface treatments improved the strength of the ceramic bond to composite resin. It had been suspected previously that an initial rough surface morphology might be important in the development of an appropriate bonding surface microstructure.[1"10] Hitherto, evaluation of the effect of the fitting surface produced on the refractory investment die had not been available. A technique for producing such refractory investment die as

Table 5. Results of the Scheffe multiple range test and a summary of the cohesive failures.

	Mean	No. of cohesive failures	Polished only P	P-Gb	Refractory only R	P-etched	P-Gb/ Etch	P-etch/sil
			Significantly different sub-groups at $p < 0.05$					
Polished	0.2276	**0**	*					
P-Gb*	4.1144	**0**	*					
Refractory	5.2313	**0**	*					
P-etched	6.2862	10	*					
P-Gb/Et*	7.8602	**5**	*					
P-Et/Si*	8.3172	**8**	*					
P-Gb/Si	8.8294	**5**	*					
P-Sil	9.3733	**9**	*					
R-Gb	10.2895	**0**	*					
P-3x	10.7171	**10**	*					
R-Gb/Et	11.7542	**5**	*	*				
R-Etch	11.8174	**8**	*	*				
R-Etch/Silane	13.7004	**10**	*	*	*	*		
R-Silane	14.5971	**9**	*	*	*	*		
R-Gb/Silane	14.7576	10	*	*	*	*		
R-3X[T]	15.9556	10	*	*	*	*	*	*

*Gb: gritblasted, Et: etched, Si: silane
ᵀ 3x refers to the triple pre-treatment of gritblasting/aciid etching/silane priming

applied in this investigation had been described.[13] A clean refractory surface was achieved in this study without having to sandblast off the refractory investment. Lack of such a clean surface apparently contributed to the problem of investigating the effect of the refractory surface on the bonding surface micro structure. The fact that the refractory fitting surfaces had higher bond strengths suggested that an initial rough surface morphology was appropriate for composite resin bonding. This was in agreement with most previous studies.[2,4,8,14] The effect of increased area of surface contact created by rough and undulated ceramic surface topography between the fluid resin and the ceramic base may be contributory to the higher bond strength. The increased total surface area available for bonding to the refractory investment die fitting surface was in contrast to a smooth fitting surface of a similar circumference. This effect was amplified in the etched surfaces.[15] Pair-wise comparisons of all the treated subgroups (excluding the controls), showed that there was no statistically significant difference in the bond strength irrespective of the type of pre-treatment of the porcelain surface. There was no significant difference between the etched surface subgroups, the silane primed subgroups and the gritblasted/etched/silane primed subgroups. The etched surface subgroups also behaved similarly suggesting that the difference in method of fitting surface production was not a major contributor to the bond strength once the ceramic fitting surfaces had been treated. The silane treated sub-groups had overall higher values than the etched sub-groups. This emphasized that silane treatment was very important for the achievement of high bond strengths.

A review of the modes of bond failure revealed these important findings. Cohesive failure within the porcelain indicated that the bond between the composite resin and the ceramic exceeded the cohesive strength of the ceramic. The trend shown by these results was similar to those from other studies.[1,8,10]. It was interesting to observe that etching alone or silane priming alone produced cohesive porcelain failure in all the samples. However, if the samples were gritblasted before either of the two treatments was carried out, equal numbers of cohesive and mixed failures were observed. Other studies[5 9 14] reported similar observations although the phenomenon had not been adequately explained. It may be reasonable to suggest mat irregular loss of material occurring at the ceramic base during gritblasting contributed to the mode of failure of these specimens; a failure starting from a weak marginal junction between the ceramic and composite resin at the interface. The failure may have originated with the fracturing off of the ceramic base because the cohesive strength of the ceramic became weaker than the interfacial bond strength. By contrast, if there was no irregularity at the marginal junction, failure took place within the ceramic base in a purely cohesive manner.

Van Noort et al, in their critique of bond strength studies, suggested that shear bond failures are more indicative of the strength between the ceramic and the composite resin at the point of impact of the testing blade rather than a true test of the interfacial bond strength.[16] This reasoning may offer an explanation for the cause of the inconsistencies in the results particularly in the modes of bond failure. A combined failure would therefore trace the path traveled by the initial cohesive failure within the porcelain, following the line of least resistance until it emerged at the interface, before the composite resin was dislodged by the testing blade. This meant that investigators might have been testing the difference between the bond strength at the point of impact of the blade and the cohesive strength as in the case of Flexoceram inlay porcelain, once cohesive failure started.

It was further suggested that all the samples in the etched only groups that failed cohesively might have been evidence of the weakening of the ceramic base from the loss of substance rather than a reflection of the interfacial bond strength. Furthermore, it was observed that once pure cohesive failure occurred it became difficult to attribute the cause to a specific type of surface treatment.

Clinical Implications

It did not seem to matter whether ceramic veneers/inlays were fabricated on platinum foil or in refractory investment die material because there was no significant difference in the bond strengths achieved with luting composite resin resulting from either surface when no further treatment was applied. The lower cost and higher bond strength to composite resin may, however, favor the choice of the refractory investment material over the more expensive platinum foil. Treatment of ceramic fitting surfaces by acid etching and silanation contributed significantly to the retention of the restoration. Silane treatment appeared to be necessary for effective composite resin bonding to the fitting surfaces of ceramic restorations.

Conclusions

1. Physical attraction was not a significant contributor to the interfacial bond strength as shown by the polished surface subgroup.
2. Macro-mechanical retention was not significant to the interfacial bond strength (untreated

refractory surface or gritblast-only subgroups).

3. Micro-mechanical adhesion contributed significantly to the porcelain/composite resin interfacial bond strength (HF-etched subgroups atp>0.05).
4. Chemical adhesion contributed significantly to the porcelain/composite resin interfacial bond strength (silane primed subgroups at pO.001).
5. A combination of the three surface treatments on one disc surface produced highly significant increases in bond strengths at p<0.001).
6. Acid etching and silane application are recommended for porcelain fitting surface treatment during the bonding of ceramic veneers, inlays and onlays to the tooth surface.

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