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ORIGINAL ARTICLE

Influence of various surface-conditioning methods on the bond strength of metal brackets to ceramic surfaces

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With the increase in adult orthodontic treatment comes the need to find a reliable method for bonding orthodontic brackets onto metal or ceramic crowns and fixed partial dentures. In this study, shear bond strength and surface roughness tests were used to examine the effect of 4 different surface conditioning methods: fine diamond bur, sandblasting, 5% hydrofluoric acid, and silica coating for bonding metal brackets to ceramic surfaces of feldspathic porcelain. Sandblasting and hydrofluoric acid were further tested after silane application. A total of 120 ceramic disc samples were produced, and 50 were used for surface roughness measurements. The glazed ceramic surfaces were used as controls. Metal brackets were bonded to the ceramic substrates with a self-curing composite. The samples were stored in 0.9% NaCl solution for 24 hours and then thermocycled (5000 times, 5°C to 55°C, 30 seconds). Shear bond tests were performed with a universal testing device, and the results were statistically analyzed. Chemical surface conditioning with either hydrofluoric acid (4.3 μm) or silicatization (4.4 μm) resulted in significantly lower surface roughness than mechanical conditioning (9.3 μm, diamond bur; 9.7 μm, sandblasting) ($P < .001$). The surface roughness values reflect the mean peak-and-valley distances. The bond strengths of the brackets bonded to the ceramic surfaces treated by hydrofluoric acid with and without silane (12.2 and 14.7 MPa, respectively), silicatization (14.9 MPa), and sandblasting with silane (15.8 MPa) were significantly higher ($P < .001$) than those treated by mechanical roughening with fine diamond burs (1.6 MPa) or sandblasting (2.8 MPa). The highest bond strength values were obtained with sandblasting and silicatization with silane or hydrofluoric acid without silane; these fulfilled the required threshold. The use of silane after hydrofluoric acid etching did not increase the bond strength. Diamond roughening and sandblasting showed the highest surface roughness; they can damage the ceramic surface. Acid etching gave acceptable results for clinical use, but the health risks should be considered. The silicatization technique has the potential to replace the other methods; yet cohesive failures were observed in the ceramic during removal of the brackets. (Am J Orthod Dentofacial Orthop 2003;123:540-6)

Adult orthodontic treatment frequently requires bonding brackets onto various types of dental restorations. Ceramics are commonly encountered as esthetic restorative materials for crowns and fixed partial dentures. The orthodontist might not know whether the dental ceramic is feldspathic porcelain, aluminous porcelain, or glass ceramic. It is very common to find feldspathic porcelain in ceramic-fused-to-metal restorations.[1] However, the bond strength of composite resins to ceramic restorations has often been reported to be insufficient.[2-4]

Numerous conditioning methods have been suggested for pretreating ceramic surfaces. Organosilane coupling agents are suggested to enhance bonding of brackets to ceramic.[2,5] Hydrofluoric (HF) acid and acidulated phosphate fluoride were reported to facilitate micromechanical retention,[4,6-8] but HF acid has been found to be a harmful and irritating compound for soft tissues. Nevertheless, the efficiency of these agents to improve the bracket bonding on ceramics has been well investigated.[1-3,9-11] Although earlier studies have relied on mechanical roughening of the ceramic surfaces, the bond strength of composite resins bonded to such ceramic restorations was unsatisfactory.[12,13] Furthermore, mechanical roughening with fine and coarse diamond burs and sandblasting are reported to provoke

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crack initiation and propagation within the ceramic.[11,14-18] Because the restorations generally remain in the mouth after debonding the brackets, damage to the ceramic due to extreme roughening of the surfaces during pretreatment or debonding must be avoided.[1,2,4,19,20]

To improve bond strengths of composite resins to ceramics, combinations of different mechanical and chemical conditioning methods are recommended.[4,8,16,21-25] Previous studies have shown that chemical conditioning methods such as silanation increase the adhesion of the composite resin bond to the ceramic.[1-3,6,14,17,26,27] The silica of the dental ceramic is chemically united with the acrylic group of the composite resin through silanation. However, the results are contradictory, showing that using silane with HF acid does not increase the bond strength.[24]

It is not always possible to make extrapolations from in vitro studies to clinical situations. One critical aspect in such studies is the difference in storage conditions.[4,5,19,20,24,25,28] Experimental specimens must be subjected to thermocycling because differences in thermal expansion coefficients and microleakage might affect the bond strength of the bracket to the ceramic. However, it is unknown whether a specific threshold of shear bond strength ensures clinical durability or increases the risk of cohesive failure.[1,17,18,24]

A recently introduced air abrasion technique, based on tribochemical silica coating, provides ultrafine mechanical retention by sandblasting, as well as a chemicophysical bonding between the composite resin and the ceramic or metal alloy by using a silane coupling agent.[29-32] The surfaces are blasted with 30 μm grain size aluminum oxide modified with silisic acid, CoJet-Sand (ESPE, Seefeld, Germany), with an intraoral sandblaster. The blasting pressure embeds silica particles in the metal or ceramic surface, rendering the surface chemically more reactive to resin via silane. Superior bond strengths were achieved with this technique onto metal crowns compared with Al_20_3 or diamond roughening only.[33] To the authors' knowledge, the silicatization procedure has not been investigated for orthodontic purposes. This system is being used for intraoral ceramic repair with satisfactory results.[34,35] It was hypothesized that if this method is efficient, the intraoral use of hazardous chemical agents or other systems based on mechanical roughening can be avoided.

The objectives of this study were to determine the surface roughness of the ceramics after various surface conditioning methods and to evaluate the shear bond strengths of metal brackets bonded to ceramic surfaces with these treatment methods alone or combined with silane.

MATERIAL AND METHODS

A total of 120 disc samples, 5 mm thick and 8 mm in diameter, were fabricated from the base alloy Wiron 88 (Bego, Bremen, Germany), consisting of 64% Ni, 24% Cr, and 10% Mo. Feldspathic porcelain (VMK68, Vita, Bad Säckingen, Germany) at a thickness of 2 mm was fired onto the alloy discs. All ceramic disc samples were glazed before they underwent conditioning. The samples were used consecutively for testing the 4 different surface conditioning methods: fine diamond bur (30 μm, Brasseler, Lemgo, Germany), sandblasting (50 μm aluminum oxide), HF acid (5%, Vita Ceram Etch, Bad Säckingen, Germany), and silica coating (CoJet-Sand). The test design and the manufacturers of the materials are given in Table I. Untreated glazed surfaces were used as controls.

The cylindrical diamond burs, with their shafts parallel to the surface of the sample, were rotated at 40,000 rpm and applied at a force of approximately 1N. Sandblasting was performed vertically from a distance of 2 cm at a pressure of 2.5 bar for 10 seconds with an intraoral sandblasting device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark). The ceramic surfaces were etched with 5% HF acid for 90 seconds in a ventilated laboratory; the technician wore acid-resistant gloves and protective glasses. The etching gel was rinsed in a polyethylene cup, and the diluted solution was neutralized with neutralizing powder and washed thoroughly for 20 seconds with oil-free water as recommended by the manufacturer. HF acid with a low concentration was chosen because of its reduced health risks.

For the silicatization process, the sandblasting device was again used but filled with CoJet-Sand. According to the manufacturer's instructions, the CoJet-Sand was blasted vertically onto the metal surfaces from a distance of approximately 10 mm, at a pressure of 2.5 bar for 13 seconds. Silane (ESPE-Sil, ESPE, Seefeld, Germany) was applied onto the conditioned samples following the protocol and allowed to dry (5 minutes) under visual control.

In an additional study, after each surface conditioning method, the surface roughness of 10 samples from the 4 main conditioning groups and the control group was measured 3 times with the Perthometer S8P 4.51 (Feinprüf GmbH, Göttingen, Germany). These samples were not used for the shear bond test because the measurements destroyed the surfaces. *Surface roughness* is defined as the mean value calculated from 5

Table I. Groups of ceramic surface treatments with and without silane application

Number	*Conditioning*	*Method, material*	*Manufacturer*	*Silane*	*Test method*
10	Control	Glazed ceramic surface			Surface roughness
10					Shear bond test
10	Mechanical	Fine diamond bur 30	Brasseler (Lemgo, Germany)		Surface roughness
10		μm			Shear bond test
10	Mechanical	Sandblasting Al_2O_3 50	Dento-Prep (RØNVIG A/S,		Surface roughness
10		μm, 10 s	Daugaard, Denmark)		Shear bond test
10				X	Shear bond test
10	Chemical	Hydrofluoric acid 5%,	Vita Ceramic Etch (Vita,		Surface roughness
10		90 s	Bad Säckingen, Germany)		Shear bond test
10				X	Shear bond test
10	Chemical	Silication Al_2O_3 +	CoJet-Sand (ESPE, Seefeld,	X	Shear bond test
10		SiO_2 30 μm	Germany)		

single roughness measurements. Each value represents the distance between the lowest and the highest points of the profile.

A total of 70 metal brackets for maxillary central incisors (Ultratrimm, Dentaurum, Pforzheim, Germany) were bonded to each conditioned ceramic surface with a self-curing composite resin (Concise, 3M, St Paul, Minn). The average surface area for the bracket base was 12.029 mm^2. The ceramic surfaces were cleaned and dried with oil-free air, the adhesive was mixed according to the manufacturer's instructions, and the composite was applied to the bracket base. The bracket was placed onto the ceramic surface with bracket pliers applying a force of about 5 N. Before the resin had set, the excess was removed around the bracket, and the test samples were stored in 0.9% NaCl solution for 24 hours. All samples were thermocycled 5000 times between 5°C and 55°C with a dwelling time of 30 seconds. The shear bond test was performed with a universal testing device (Zwick 1120, Ulm, Germany). The ceramic discs were mounted in a jig with the brackets vertical. The shear force at a crosshead speed of 1 mm/minute was transmitted to the bracket by a square plate the same size as the bracket. The force required to shear the bracket was recorded, and the bond strengths were calculated in megapascals (MPa). The sheared surfaces were further observed to determine the mode of the bond failure.

The results were statistically analyzed by using nonparametric methods (Kruskal-Wallis and Mann-Whitney tests) and corrected with the Bonferroni adjustment because of the significance levels.

RESULTS

Among the conditioning groups, significantly higher ($P < .001$) surface roughness values were obtained with the diamond bur (9.3 μm) and sandblasting (9.7 μm) than with HF acid application (4.3 μm) and silicatization (4.4 μm); the values for the control group were the lowest (Fig 1, Table II).

Bond strengths of the brackets to the ceramic surfaces treated by HF acid with and without silane (12.2 and 14.7 MPa, respectively), silicatization (14.9 MPa), and sandblasting with silane (15.8 MPa) were significantly higher ($P < .001$) than those treated by mechanical roughening with a fine diamond bur (1.6 MPa) or sandblasting (2.8 MPa) (Fig 2, Tables III and IV). The brackets bonded to the glazed ceramic surfaces failed during thermocycling.

Adhesive failures between the ceramic and the composite resin were observed mainly in the diamond bur group, with HF acid application with and without silane, and with sandblasting without silane. All failures were cohesive within the ceramic layer after silicatization and silanation, but the failure rates were 70% cohesive and 30% adhesive in the groups of sandblasting with silane. No cohesive failures were observed in the composite resin in any group.

DISCUSSION

The aim of this study was to find the most reliable method for bonding metal brackets onto metal or ceramic crowns or fixed partial dentures. Although it is difficult to apply the results of in vitro studies to clinical practice, it has been suggested that bond strengths of 6 to 10 MPa are sufficient for orthodontic bracket bond-

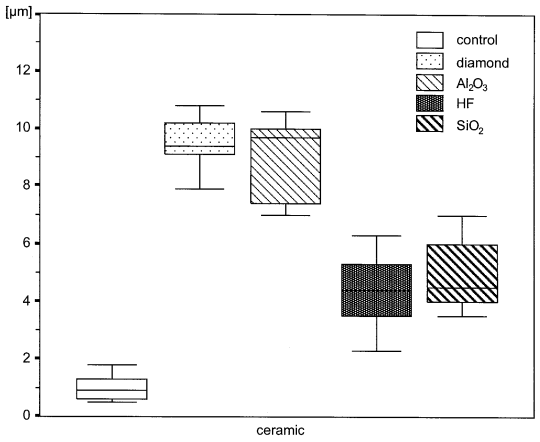

Fig 1. Surface roughnesses of tested ceramic surfaces after conditioning methods (µm). *Control*, glazed ceramic; *diamond*, diamond bur; *Al2O3*, sandblasting; *HF*, hydrofluoric acid; *SiO2*, silicatization. Results presented as boxplots. Horizontal line in middle of each boxplot shows mean value; horizontal lines in box give 25% and 75% quartiles; lines outside box give 5% and 95% quartiles.

ing.[2,3,19,36-38] Yet the direct transfer of this value to clinical situations is not universally accepted because the bracket-ceramic bond is influenced by many environmental factors.[1,24,39,40] Even though the clinical relevance of in vitro studies is limited, they are essential in testing new methods before they can be used in vivo.

In this study, HF acid etching and silicatization and sandblasting followed by silane application fulfill the required threshold. The results for HF acid etching and sandblasting followed by silane application agree with those of others.[4,12,13,16,21,23,24,40]

Thermocycling of at least 500 cycles is required to test the bond strength of brackets to ceramics,[1,28,41] because of artificial aging and also the different thermal expansion coefficients of ceramic, resin, and metal. The temperature change could also contribute to water contamination at the bond interface and weaken the resin over a long time. Usually, thermocycling has a significant effect on the bond strength: the bond values decrease[1,12,20,21,25,37,40] in comparison with the studies in which no thermocycling was applied.[2,3,6,16,17,23,26] Most of the studies involved different thermocycling times, ranging from 1005,13 to 5000 cycles.[34,41] Moreover, the specimens were kept in deionized water for different times before testing bond strengths[2,3,8,16,17,38,39,42-44]; this makes it difficult to make direct comparisons between studies.

Shear bond testing after thermocycling has been the standard method of measuring the bond strength of brackets to different substrates.[1,4,45] However, the effect of shear bond testing must be questioned because nonuniform stress distribution is generated.[5,38,46] Thus it is not surprising that failures can occur cohesively.[45]

Table II. Significant differences between surface roughnesses of ceramic after different conditioning methods

	Control	*Diamond*	*Al_2O_3*	*HF*	*Silicatization*
Control		***	***	***	***
Diamond	***		NS	***	***
Al_2O_3	***	NS		***	***
HF	***	***	***		NS
Silicatization	***	***	***	NS	

***$P < .001$.
NS, not significant.

Cohesive failures within the ceramic could be interpreted so that the composite resin-ceramic compound was stronger than the ceramic layer itself.[11,39] In the silicated group, the interface reached the maximum of the required bond strength; yet such a fracture mode resulted in damage to the ceramic surface—a clinical disadvantage. Adhesive failures are preferred to avoid ceramic fractures during debonding.[13] It was reported that if bond strengths between the ceramic and the composite resin were higher than 13 MPa, the fracture would be cohesive.[21] The bond strength obtained in the silicatization group exceeded this value and exhibited cohesive failures in the ceramic, whereas the bond strengths in the groups of HF acid and sandblasting followed by silane application also had higher values but with mainly adhesive failures.

In clinical practice, the incidence of ceramic damage while debonding the brackets was stated to be very low or not to occur at all, and to be independent of the previously used bonding method.[1,24] The reason for this discrepancy might be that clinically proper and safe debonding techniques with adequate peeling forces are different from shear testing in the laboratory.[2,4,18,38,47] However, the possibility of porcelain fractures cannot be excluded.[20,26,38,45] Therefore, it is impossible to predict whether the risk of ceramic damage will be higher in clinical conditions when the silica coating method is used.

Other factors influencing cohesive failure in the ceramic are the bonding agent,[3,4,38] the ceramic type, and the surface treatment, eg, deglazing.[15,18,39] Further studies with other luting agents with different silanes should be made.

In this study, the glazed ceramic surfaces were used as control groups to represent the clinical situation.

Table III. Significant differences between shear bond strength values of brackets regarding the several conditioning methods of the ceramic

	Control	*Diamond*	*Al_2O_3*	*Al_2O_3 + silane*	*HF*	*HF + silane*	*SiO_2 + silane*
Control		***	***	***	***	***	***
Diamond	***		NS	***	***	***	***
Al_2O_3	***	NS		***	***	***	***
Al_2O_3 + silane	***	***	***		NS	*	NS
HF	***	***	***	NS		NS	NS
HF + silane	***	***	***	*	NS		NS
SiO_2 + silane	***	***	***	NS	NS	NS	

*$P < .05$; ***$P < .001$.
NS, not significant

Table IV. Shear bond strength values (MPa) of brackets to conditioned ceramic surfaces

	Mean	*SD*
Control	0	0
Diamond	1.6	0.8
Al_2O_3	2.8	1.5
Al_2O_3 + silane	15.8	4.2
HF	14.7	3.3
HF + silane	12.2	3.4
SiO_2 + silane	14.9	3.8

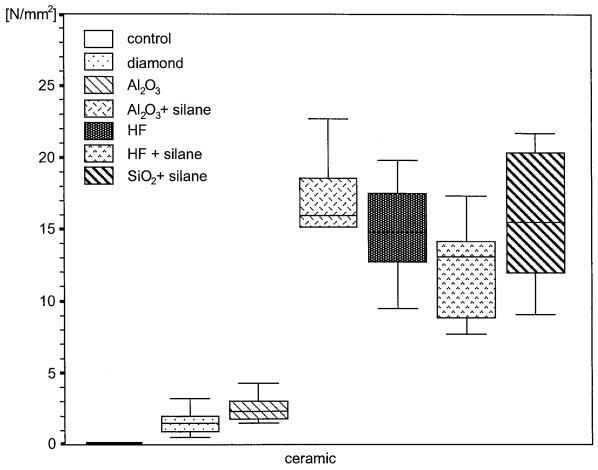

Fig 2. Shear bond strengths (MPa) of metal brackets bonded to conditioned ceramic discs. *Control*, glazed ceramic; *diamond*, diamond bur; *Al2O3*, sandblasting; *Al2O3 + silane*, sandblasting and silanation; *HF*, hydrofluoric acid; *HF + silane*, hydrofluoric acid and silanation; *SiO2 + silane*, silicatization and silanation.

Surface conditioning was performed on glazed surfaces because the highest incidence of ceramic fracture associated with debonding was reported to occur on the deglazed, silanated, or roughened surfaces.[17,18,37] It was further shown that the removal of surface glaze by grinding reduces the transverse strength of the porcelain to half that when glaze was present, and that the glaze effectively reduces crack propagation.[48] The cracks that begin during deglazing, eg, by sandblasting, lead to ceramic damage while debonding.[15] Recommendations were given to avoid deglazing the ceramic surface and to prefer methods that provide a sufficient bond with less roughening.[16,17,20,37]

There is also widespread agreement in the literature that roughening the surface is a prerequisite for achieving sufficient bond strength on metal or ceramic.[1,4,13,21] Many authors have recommended using an intraoral sandblaster for surface roughening.[1,12,23,38,49] In this study, air abrasion with aluminum oxide exhibited higher bond strengths than roughening with diamond burs; this agrees with previous reports.[1,4,38] Such a relationship was also observed in mechanical retention after bonding brackets on metal restorations.[33] The high surface roughness caused by sandblasting was a disadvantage, even though the bond strength was high. The rough ceramic surface should be polished after debonding the brackets; this is possible with ceramic polishing kits and diamond polishing pastes.[4,45] Comparable bond strengths were also achieved without creating high surface roughness with HF acid and silicatization.

Some authors have reported superior bond strength using HF acid with silane,[2,9,12,18,26,39] but our findings did not show that silane application increases the bond strength when it is used in conjunction with HF acid. However, a significant increase was noted when silane was used after sandblasting.[1,2,13,18] The silane treatment was often mentioned as essential for achieving chemical adhesion between the ceramic and the composite resin,[2,5,12,18,25,38,42] but the possible effect of thermocycling weakening the silane bond after HF acid etching must be considered.[21,24,40]

Although it is not recommended for intraoral use,

HF acid etching gel for orthodontic purposes is widely used when a ceramic restoration is involved.[3,25,40] Extreme care should be taken during intraoral application of HF acid because contact between the acid and soft tissues can cause severe tissue irritation.[6,27] Although the bond result obtained with HF acid is satisfactory, one might prefer alternative conditioning techniques because of this potential danger. Therefore, the newly introduced methods based on silicatization could be considered for ceramic surface conditioning before bracket bonding. Our findings using silicatization agree with those of previous investigations when this method is used for other purposes.[29-32,34,35]

The present study did not find an ideal surface conditioning technique for bonding metal brackets to ceramic surfaces. The use of HF acid would still be appropriate if its intraoral hazardousness is accepted. The alternative methods of sandblasting and silica coating followed by silanation produced high surface roughness and ceramic damage during debonding of the bracket, respectively. This failure mode under laboratory conditions might not have clinical significance; therefore, further clinical trials are needed to obtain experience with the silicatization technique.

CONCLUSIONS

Within the limitations of this study, the following conclusions were made:

1. Roughening the ceramic surfaces with either diamond bur or sandblasting created higher surface roughness than HF acid etching or silicatization. For surface roughness, HF acid etching and silica coating should be preferred.
2. Although the use of silane combined with HF acid etching did not increase the bond strength significantly, it increased the results in sandblasting.
3. Silicatization resulted in the most favorable bond strength among all methods tested, yet with cohesive failures in ceramic after debonding.
4. Silica coating followed by silanation might have the potential to replace the alternative methods.

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