

# Effects of surface-conditioning methods on shear bond strength of brackets bonded to different all-ceramic materials

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**SUMMARY** The aims of this study were to investigate the effects of two surface-conditioning methods on the shear bond strength (SBS) of metal brackets bonded to three different all-ceramic materials, and to evaluate the mode of failure after debonding. Twenty feldspathic, 20 fluoro-apatite, and 20 leucite-reinforced ceramic specimens were examined following two surface-conditioning methods: air-particle abrasion (APA) with 25 µm Al<sub>2</sub>O<sub>3</sub> and silica coating with 30 µm Al<sub>2</sub>O<sub>3</sub> particles modified by silica. After silane application, metal brackets were bonded with light cure composite and then stored in distilled water for 1 week and thermocycled (×1000 at 5–55°C for 30 seconds). The SBS of the brackets was measured on a universal testing machine. The ceramic surfaces were examined with a stereomicroscope to determine the amount of composite resin remaining using the adhesive remnant index. Two-way analysis of variance, Tukey's multiple comparison test, and Weibull analysis were used for evaluation of SBS.

The lowest SBS was with APA for the fluoro-apatite ceramic (11.82 MPa), which was not significantly different from APA for the feldspathic ceramic (13.58 MPa). The SBS for the fluoro-apatite ceramic was significantly lower than that of leucite-reinforced ceramic with APA (14.82 MPa). The highest SBS value was obtained with silica coating of the leucite-reinforced ceramic (24.17 MPa), but this was not significantly different from the SBS for feldspathic and fluoro-apatite ceramic (23.51 and 22.18 MPa, respectively). The SBS values with silica coating showed significant differences from those of APA. For all samples, the adhesive failures were between the ceramic and composite resin. No ceramic fractures or cracks were observed. Chairside tribochemical silica coating significantly increased the mean bond strength values.

## Introduction

The need to bond orthodontic brackets to a variety of dental restorations has become more common as the number of adult patients seeking orthodontic treatment steadily increases. Patients are increasingly demanding dental restorations that are both aesthetic and functional. Manufacturers have introduced numerous all-ceramic alternatives (Donovan, 2008). Therefore, the orthodontist is often confronted with the challenge of effectively bonding orthodontic brackets to different ceramic restorations.

The approaches suggested for bonding orthodontic brackets to ceramic restorations can be classified into three categories: mechanical, chemical, or a combination (Abu Alhaja and Al-Wahadni, 2007). Mechanical alteration of porcelain surfaces to increase bond strength has been achieved by air-particle abrasion (APA; Zachrisson *et al.*, 1996; Cochran *et al.*, 1997; Kocadereli *et al.*, 2001) or by applying coarse diamond stones (Gillis and Redlich, 1998; Abu Alhaja and Al-Wahadni, 2007). Peterson *et al.* (1998) pointed out that mechanical roughening with diamond burs and APA provokes crack initiation on the ceramic surface.

Following orthodontic treatment, ceramic restorations normally remain in the mouth, thus damage to the ceramic due to roughening during surface conditioning should be minimized (Schmage *et al.*, 2003).

Chemical alteration of the porcelain surface can be achieved by either etching or changing the porcelain bonding affinity to adhesive materials (Abu Alhaja and Al-Wahadni, 2007). Etching the porcelain surface with hydrofluoric acid (HFA) to increase bond strength has been advocated (Zachrisson *et al.*, 1996; Cochran *et al.*, 1997; Bourke and Rock, 1999; Kocadereli *et al.*, 2001; Abu Alhaja and Al-Wahadni, 2007). Clinically, there are drawbacks with the use of HFA (Abu Alhaja and Al-Wahadni, 2007). Due to the acidic and extremely corrosive nature and the capability of causing severe trauma to soft tissues and tooth substance, HFA has to be used with great care (Hayakawa *et al.*, 1992).

Silane coupling agents, which are widely accepted as adhesion promoters in clinical practice enhance the bond strength by increasing the chemical bond between the resin composite and ceramic material (Wood *et al.*, 1986; Kao

and Johnston, 1991; Cochran *et al.*, 1997; Chung *et al.*, 1999; Huang and Kao, 2001; Kocadereli *et al.*, 2001; Schmage *et al.*, 2003).

Another abrasive method is tribochemical silica coating, which provides micromechanical retention and sites for chemical adhesion. The alumina particles that are chemically silica coated are blasted onto the metal surfaces (such as base metal, noble metal alloys, or titanium) and onto the polymeric resin composite or ceramic surfaces (Matinlinna and Vallittu, 2007b). The particles form a reactive silica layer on the substrate, and, thereafter, silane must be applied for chemical bonding with a resin-based system (Hansson and Moberg, 1993).

There are several types of porcelain for ceramic restorations: silica-based ceramics (e.g. feldspathic, fluoroapatite, leucite-reinforced glass ceramics, lithium disilicate glass ceramics), glass-infiltrated or densely sintered aluminium oxide ceramics, and zirconium oxide ceramics (Blatz *et al.*, 2003; Karan *et al.*, 2007). However, only a limited number of studies exist concerning the bond strength of orthodontic brackets to different all-ceramic restorations (Chay *et al.*, 2005; Türk *et al.*, 2006; Abu Alhaja and Al-Wahadni, 2007; Karan *et al.*, 2007; Kukiattrakoon and Samruajbenjakul, 2010).

The objectives of this study were to observe the outcomes of two different surface-conditioning methods (sandblasting and tribochemical silica coating) on the shear bond strength (SBS) of metal orthodontic brackets to three different silica-based all-ceramic restorative materials (feldspathic, fluoroapatite, and leucite reinforced), and to evaluate the mode of failure after debonding.

## Materials and methods

A *post hoc* power calculation showed that for a power of 0.99, a sample of 20 ceramic specimens for each group would be required. Forty feldspathic (Vitadur Alpha; Vita Zahnfabrik, Bad Säckingen, Germany), 40 fluoro-apatite (IPS Eris layering material; Ivoclar Vivadent, Schaan, Liechtenstein), and 40 leucite-reinforced (IPS Empress Esthetic; Ivoclar Vivadent) ceramic specimens with a diameter of 6 mm and a thickness of 3 mm were fabricated and glazed according to the manufacturers' recommendations. The specimens were embedded in autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer Ltd, Newbury, Berkshire, UK) with their glazed surfaces facing upwards. For each all-ceramic material, the specimens were randomly divided into two groups according to random number tables. Each group consisted of 20 specimens and two different surface-conditioning methods were used. In the first group, APA was performed using 25 µm aluminium trioxide (Al<sub>2</sub>O<sub>3</sub>) with an air abrasion device (Bego TopTec; Bego, Germany) at a distance of approximately 10 mm and a pressure of 2.5 bars for 4 seconds. In the second group, the surfaces were treated with APA with 30 µm Al<sub>2</sub>O<sub>3</sub> particles

modified by silica (CoJet Sand; 3M-Espe, Seefeld, Germany), using an intraoral device (Microetcher; Danville Eng., San Ramon, California, USA). APA was used with a nozzle distance of approximately 10 mm from the surface at an angle of 90 degrees for 4 seconds at 3 psi.

Subsequently, silane and the adhesive primer (Transbond XT; 3M Unitek, Monrovia, California, USA) were applied to all roughened specimens. The light cure adhesive paste (Transbond XT; 3M Unitek) was applied to the mesh base of a maxillary central incisor bracket (Gemini bracket; 3M Unitek). The bracket was then seated and positioned manually on the ceramic surface. Excess composite was carefully removed from the periphery of the bracket base with an explorer. The surface-conditioning methods and the placement of the brackets were performed by two operators, YSS and TT, respectively. The adhesive paste was cured for a total of 20 seconds from two directions using a visible light-curing unit (Hilux 200; Benlioglu Dental Inc., Ankara, Turkey) with an output of 600 mW/cm<sup>2</sup>. All specimens were stored in distilled water at 37 ± 2°C for 1 week. The specimens were thermocycled 1000 times between 5 and 55°C with a dwelling time of 30 seconds. The shear bond test was performed with a universal testing device (Lloyd LRX; Lloyd Instruments Ltd, Fareham, Hants, UK) at a crosshead speed of 1 mm/minute. The bond strengths were calculated in megapascals (MPa).

The ceramic surfaces were examined with a stereomicroscope (Stemi 2000-C; Carl Zeiss, Göttingen, Germany) at a magnification of ×10 to determine the amount of composite resin remaining according to the adhesive remnant index (Årtun and Bergland, 1984) and to assess the damage to the ceramic which may have occurred during shear bond testing.

To evaluate the effect of surface-conditioning methods on the ceramic surfaces, three additional feldspathic, three fluoro-apatite ceramic, and three leucite-reinforced ceramic specimens were prepared and glazed. The surfaces of two specimens of each ceramic were then conditioned with the same experimental protocol described above. Two roughened specimens for each ceramic were gold sputtered with a sputter coater (S150B; Edwards, Crawley, Sussex, UK) and examined under a field emission scanning electron microscope (SEM, JSM-6335F; Jeol, Tokyo, Japan) at 20.0 kV. The SEM photomicrographs were taken at ×500 magnification for visual inspection.

Two-way analysis of variance (ANOVA) was used to determine significant differences among all-ceramic materials and surface-conditioning methods and their interactions. All treatment combination means for bond strength values were compared using the Tukey's multiple comparison test ( $P < 0.05$ ).

A Weibull analysis was performed, and the Weibull modulus, characteristic bond strength, correlation coefficient, and the stress levels at 5 and 10 per cent probability of failure were calculated.

## Results

The results of the two-way ANOVA are shown in Table 1. The main effects were significant differences for the surface-conditioning methods and all-ceramic materials on the SBS values ( $P < 0.001$ ; Table 1). The interaction between the conditioning methods and ceramic materials was not significant ( $P > 0.05$ ; Table 1).

The mean SBS, minimum and maximum values, and standard deviations (SDs) for each group are presented in Table 2. For each group, box plots of the SBSs are presented in Figure 1. The results of the Tukey's multiple comparison test for the mean SBS values are given in Table 2.

The lowest SBS was with APA for the fluoro-apatite ceramic (11.82 MPa), which was not significantly different from APA for the feldspathic ceramic (13.58 MPa). The SBS for the fluoro-apatite ceramic was significantly lower than for the leucite-reinforced ceramic with APA (14.82 MPa).

The highest SBS values were obtained with silica coating of the leucite-reinforced ceramic (24.17 MPa), but these were not significantly different from the SBS for feldspathic and fluoro-apatite ceramic (23.51 and 22.18 MPa, respectively). The SBS values obtained with silica coating showed significant differences from that obtained with the APA.

The parameters of the Weibull analysis for each group are given in Table 1. The Weibull distribution plots of the

probability of failure at a certain shear stress level for the two groups are depicted in Figure 2.

The modes of bond failure for the brackets after different surface-conditioning methods are given in Table 3. Adhesive failures between the ceramic and adhesive resin were found in all groups. No cracks or fractures of the ceramic surfaces were observed.

Scanning electron photomicrographs of the feldspathic, fluoro-apatite, and leucite-reinforced ceramic surfaces conditioned using different methods are presented in Figure 3. APA with 25  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles demonstrated slightly more prominent irregularities and deeper erosions (Figure 3A, 3C, and 3E). However, the silica-coating procedure created superficial irregularities and shallow erosions on the surface. The white spots, observed on the surface, were silica (Figure 3B, 3D, and 3F).

## Discussion

The results of the present study confirm that surface-conditioning methods and the type of the all-ceramic materials affect the bond strength of metal orthodontic brackets to ceramic surfaces. These results support previously published studies on this topic (Türk *et al.*, 2006; Abu Alhaija and Al-Wahadni, 2007; Karan *et al.*, 2007; Kukiattrakoon and Samruajbenjakul, 2010).

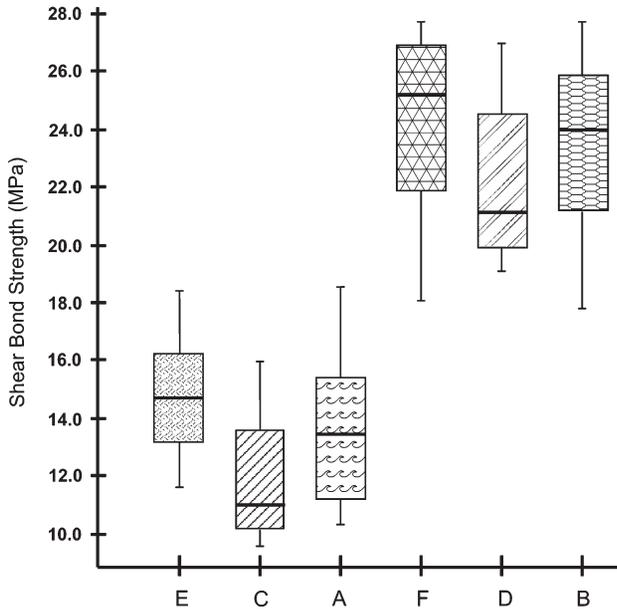
**Table 1** Two-way analysis of variance of force (Megapascals) required to debond metal brackets from dental ceramic.

Source of variation	Mean square	df	Sum of squares	F ratio	Significance
Ceramic	63.410	2	126.820	9.229	0.000
Surface conditioning	2929.543	1	2929.543	426.396	0.000
Ceramic $\times$ surface conditioning	2.564	2	5.127	0.373	0.689
Error	6.870	114	783.233		
Corrected total	3844.724	119			

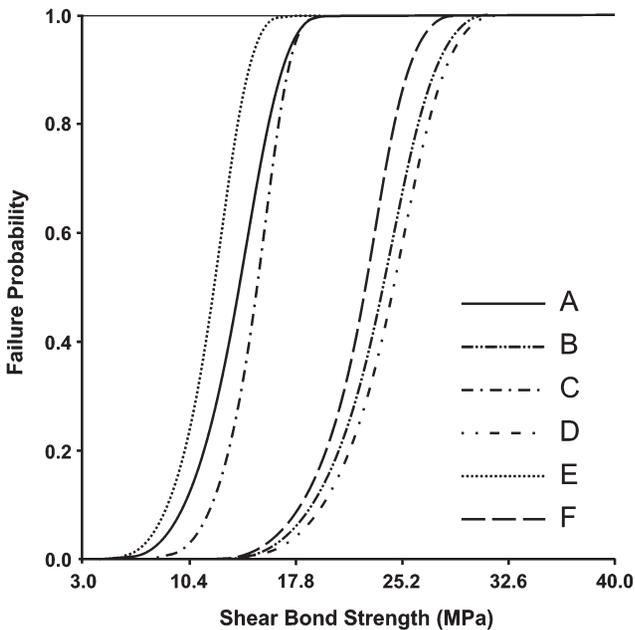
**Table 2** Mean shear bond strengths, standard deviations (SDs), minimum (Min) and maximum (Max) values, and Weibull parameters for each group ( $n = 20$ ).

Groups	Mean*	SD	Min	Max	Weibull analysis					
					Weibull modulus	Correlation coefficient	Characteristic bond strengths (MPa)	Shear stress at the 5% probability of failure (MPa)	Shear stress at the 10% probability of failure (MPa)	
Air-particle abrasion	Feldspathic	13.58 AB	2.56	10.36	18.51	4.95	0.91	16.82	9.23	10.68
	Fluoro-apatite	11.82 A	2.06	9.61	15.97	5.12	0.85	14.57	8.16	9.40
	Leucite reinforced	14.82 B	1.99	11.63	18.39	7.05	0.93	17.28	11.33	12.55
Silica-coating	Feldspathic	23.51 C	3.11	17.82	27.70	7.17	0.98	27.33	18.07	19.97
	Fluoro-apatite	22.18 C	2.71	19.10	26.93	7.30	0.86	25.76	17.14	18.93
	Leucite reinforced	24.17 C	3.08	18.09	27.70	7.46	0.98	27.94	18.76	20.66

\*Means for groups having the same letters show homogeneous subsets,  $\alpha = 0.05$



**Figure 1** Box plot of the distribution of the shear bond strengths: (A) Feldspathic ceramic—air-particle abrasion (APA) with 25 µm Al<sub>2</sub>O<sub>3</sub>, (B) Feldspathic ceramic—silica coating with 30 µm SiO<sub>2</sub>, (C) Fluoro-apatite ceramic—APA with 25 µm Al<sub>2</sub>O<sub>3</sub>, (D) Fluoro-apatite ceramic—silica coating with 30 µm SiO<sub>2</sub>, (E) Leucite-reinforced ceramic—APA with 25 µm Al<sub>2</sub>O<sub>3</sub>, (F) Leucite-reinforced ceramic—silica coating with 30 µm SiO<sub>2</sub>.



**Figure 2** Cumulative failure probabilities versus shear bond strengths. (A) Feldspathic ceramic—air-particle abrasion (APA) with 25 µm Al<sub>2</sub>O<sub>3</sub>, (B) Feldspathic ceramic—silica coating with 30 µm SiO<sub>2</sub>, (C) Fluoro-apatite ceramic—APA with 25 µm Al<sub>2</sub>O<sub>3</sub>, (D) Fluoro-apatite ceramic—silica coating with 30 µm SiO<sub>2</sub>, (E) Leucite-reinforced ceramic—APA with 25 µm Al<sub>2</sub>O<sub>3</sub>, (F) Leucite-reinforced ceramic—silica coating with 30 µm SiO<sub>2</sub>.

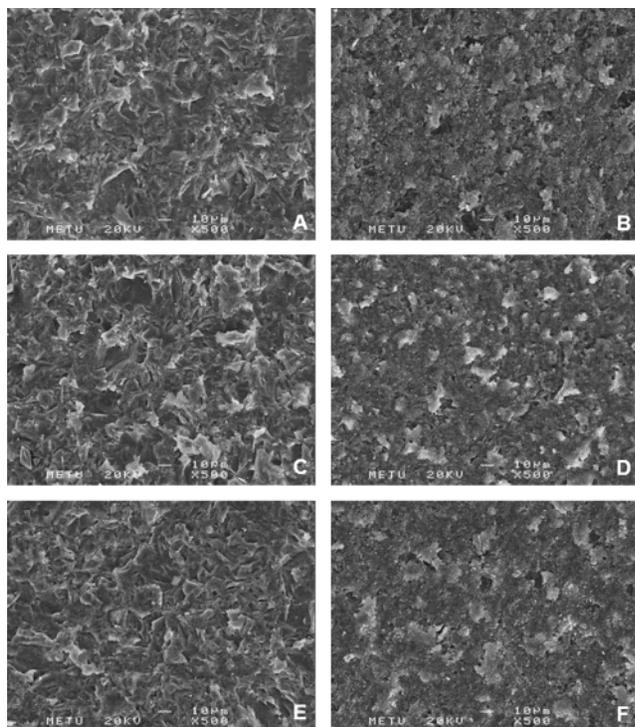
**Table 3** Modes of failure of metal brackets bonded to two all-ceramics after two surface-conditioning methods.

Groups		Adhesive remnant index score			
		0	1	2	3
Air-particle abrasion	Feldspathic	20	—	—	—
	Fluoro-apatite	20	—	—	—
	Leucite reinforced	20	—	—	—
Silica coating	Feldspathic	20	—	—	—
	Fluoro-apatite	20	—	—	—
	Leucite reinforced	20	—	—	—

Score 0 = no composite left on ceramic surface; score 1 = less than half of composite left; score 2 = more than half of composite left; score 3 = all composite left on ceramic surface.

Glazed porcelain surfaces are not amenable to resin penetration for orthodontic bonding (Smith *et al.*, 1988). For successful bonding of orthodontic brackets to porcelain, mechanical or chemical removal of the glazed surface is essential to obtain mechanical interlocking.

A tensile bond strength value of 6–8 MPa would be adequate to resist treatment forces (Reynolds, 1975). In this study, the SBS values obtained with APA were above this clinically acceptable level. Nevertheless, silica coating showed higher SBS values than APA. APA is used to roughen the ceramic surface to increase the amount of bonding area and mechanical locking. Silica-coating systems are used to produce a silica layer on the ceramic surface with the aid of the high-speed surface impact of the alumina particles modified by silica followed by silanization (Della Bona, 2005). The tribochemical effect of the silica-coating system may be explained by two bonding mechanism 1: the creation of a topographic surface allowing for micromechanical bonding to resin and the chemical bond of the silica-coated ceramic surface, the silane agent, and the resin material (Della Bona, 2005). Sun *et al.* (2000) reported that the components of the blasting abrasive can penetrate into the metal to a depth of 15 µm. The third step of tribochemical system is to apply silane on the treated surfaces. After silica coating, the silica layer on the ceramic surface provides a base for silane. An immediate silane application forms covalent bonding between the silica-coated ceramic layer and resin composite (Matinlinna and Vallittu, 2007a). Moreover, the silane also contributes to the improved surface wettability to resin (Thurmond *et al.*, 1994; Della Bona *et al.*, 2004). For enhanced clinical success, the use of a silane coupling agent for creating long-term bonds of resin to ceramic has been suggested (Matinlinna and Vallittu, 2007a). All these findings may explain the higher SBSs with silica coating compared with APA.



**Figure 3** Scanning electron photomicrographs: (A) Feldspathic ceramic—air-particle abrasion (APA) with 25  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , (B) Feldspathic ceramic—silica coating with 30  $\mu\text{m}$   $\text{SiO}_2$ , (C) Fluoro-apatite ceramic—APA with 25  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , (D) Fluoro-apatite ceramic—silica coating with 30  $\mu\text{m}$   $\text{SiO}_2$ , (E) Leucite-reinforced ceramic—APA with 25  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , (F) Leucite-reinforced ceramic—silica coating with 30  $\mu\text{m}$   $\text{SiO}_2$ . Original magnification  $\times 500$  and bar = 10  $\mu\text{m}$ .

Karan *et al.* (2007) found that the highest SBS values were achieved with silica coating on three different ceramics (feldspathic, lithium disilicate, and leucite-reinforced). They concluded that silica-coating could replace other conditioning techniques in bonding brackets to ceramic. Ozcan *et al.* (2004) also observed that brackets treated with silica coating had significantly greater bond strengths on tested feldspathic porcelain.

When the results of the study are considered in terms of the type of the all-ceramics, both surface treatments increased bond strength. There was no statistical difference between the veneering materials, feldspathic, and fluoro-apatite ceramics, but bond strength values were higher for leucite-reinforced ceramic. This was probably due to microstructural differences, processing techniques, and the more porous structure of the veneering materials. However, leucite-reinforced ceramic is processed by heat-press techniques and is based on extremely homogeneous and increased density of the crystals (Oh and Shen, 2003, 2005; Shen *et al.*, 2004).

For all samples, adhesive failures between the ceramic and composite resin were observed in the present study. This type of adhesive failure demonstrates that the bond strength between the composite and bracket and the cohesive strength

of the composite was stronger than the bond strength between the composite and ceramic. Adhesive failures at the ceramic/composite interface are preferred to avoid ceramic fractures during debonding (Smith *et al.*, 1988).

The Weibull analysis conveys information concerning the probability of bracket failure and presents the orthodontist with an indication of how the material or bracket is likely to perform in a clinical situation, i.e. the oral environment (Fox *et al.*, 1994). Littlewood *et al.* (2001) suggested using the 5 per cent chance of failure as a more appropriate level to assess bond strength. According to those authors, the bond strength of a material with a 5 per cent chance of failure should be at least 5.4 MPa. In the present study, SBS showed shear stress levels higher than 5.4 MPa at the 5 per cent probability of failure for all groups. This result suggests acceptable SBS for all groups in the oral environment. Nevertheless, as with any *in vitro* study, discretion should be exercised when attempting to extrapolate laboratory findings to the clinical setting.

## Conclusions

1. Chairside tribochemical silica coating significantly increased mean bond strength values.
2. With all surface-conditioning methods, leucite-reinforced ceramic, in general, showed a higher SBS than feldspathic and fluoro-apatite ceramics.
3. For all samples, the adhesive failures were between the ceramic and composite resin. No ceramic fractures or cracks were observed.

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