Factors affecting the shear bond strength of metal and ceramic brackets bonded to different ceramic surfaces

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SUMMARY The aims of this study were to evaluate the shear bond strength (SBS) of metal and ceramic brackets bonded to two different all-ceramic crowns, IPS Empress 2 and In-Ceram Alumina, to compare the SBS between hydrofluoric acid (HFA), phosphoric acid etched, and sandblasted, non-etched all-ceramic surfaces. Ninety-six all-ceramic crowns were fabricated resembling a maxillary left first premolar. The crowns were divided into eight groups: (1) metal brackets bonded to sandblasted 9.6 per cent HFA-etched IPS Empress 2 crowns; (2) metal brackets bonded to sandblasted 9.6 per cent HFA-etched In-Ceram crowns; (3) ceramic brackets bonded to sandblasted 9.6 per cent HFA-etched IPS Empress 2 crowns; (4) ceramic brackets bonded to sandblasted 9.6 per cent HFA-etched IPS Empress 2 crowns; (5) metal brackets bonded to sandblasted 37 per cent phosphoric acid-etched IPS Empress 2 crowns; (6) metal brackets bonded to sandblasted 37 per cent phosphoric acid-etched In-Ceram crowns; (7) metal brackets bonded to sandblasted, non-etched IPS Empress 2 crowns; and (8) metal brackets bonded to sandblasted, non-etched In-Ceram crowns. Metal and ceramic orthodontic brackets were bonded using a conventional light polymerizing adhesive resin. An Instron universal testing machine was used to determine the SBS at a crosshead speed of 0.1 mm/minute. Comparison between groups was performed using a univariate general linear model and chi-squared tests.

The highest mean SBS was found in group 3 (120.15 ± 45.05 N) and the lowest in group 8 (57.86 ± 26.20 N). Of all the variables studied, surface treatment was the only factor that significantly affected SBS (P < 0.001). Acid etch application to sandblasted surfaces significantly increased the SBS in groups 1, 2, 5, and 6. The SBS of metal brackets debonded from groups 1, 3, and 5 were not significantly different from those of groups 2, 4, and 6. All debonded metal brackets revealed a similar pattern of bond failure at the adhesive–restorative interface. However, ceramic brackets had a significantly different adhesive failure pattern with dominant failure at the adhesive–bracket interface. Ceramic fractures after bracket removal were found more often in groups 1–4. No significant difference in ceramic fracture was observed between the IPS Empress 2 and In-Ceram groups.

Introduction

Most adult patients usually present with restored teeth. Dental ceramic is widely used to restore missing or damaged teeth. Various types of ceramics have been developed. These vary in chemical composition, method of manufacture, and physical properties. All-ceramic restorations, also known as 'free metal restorations', are among the most recent types of ceramics. These include conventional powder and slurry, castable, machinable, pressable, and infiltrated ceramics (Rosenblum and Schulman, 1997). The IPS Empress system (Ivoclar-Vivadent, Şchaan, Liechtenstein) is supplied in a form of feldspathic ingots, which are made up of microleucite crystals that are produced by controlled crystallization in a glass containing nucleating agents. These ingots are heated and subsequently pressed in a mould using an alumina plunger to form an all-ceramic restoration.

In-Ceram ceramics (Vita Zahnfabrik, H. Rauter GmbH & Co. KG, Bad Säckingen, Germany) are fabricated by an infiltrated molten glass matrix in a porous core composed of aluminium oxide or spinel. The glass-infiltrated core is

subsequently veneered with feldspathic porcelain. Restorations fabricated by aluminium oxide infiltrated cores are considered the strongest all-ceramic restoration (Rosenblum and Schulman, 1997).

Ceramic is an inert material. It does not adhere chemically to any of the currently available bonding resins. Conventional acid etching is ineffective in the preparation of ceramic surfaces for mechanical retention of brackets and orthodontic attachments (Zachrisson et al., 1996). It is important to prepare ceramic surfaces prior to bonding. Numerous approaches have been reported in the literature. These can be classified into three major groups, namely mechanical, chemical, or a combination. Mechanical alteration of porcelain surfaces to increase bond strength has been achieved by sandblasting (Zachrisson et al., 1996; Cochran et al., 1997; Kocadereli et al., 2001). However, it has been shown that although roughening of porcelain surfaces significantly increases bond strength, it also results in a higher incidence of porcelain fracture associated with debonding (Kao et al., 1988).

Numerous types of acid etching solution with variable concentrations have been developed. These include hydrofluoric acid (HFA) gel (Zachrisson *et al.*, 1996; Kocadereli *et al.*, 2001), acidulated phosphate fluoride (APF; Major *et al.*, 1995), and phosphoric acid gel and solutions (Yen *et al.*, 1993). The most commonly used ceramic acid etchant is a 9.6 per cent HFA gel (Stangel *et al.*, 1987). A 2–4 minute application of HFA gel on ceramic surface has been advocated (Zachrisson *et al.*, 1996; Zachrisson, 2000). However, HFA is a strong acidic solution that should be applied with extreme caution avoiding contact with the soft tissues (Zachrisson *et al.*, 1996; Bourke and Rock, 1999; Larmour *et al.*, 2006; Turk *et al.*, 2006).

Due to the potential toxicity of HFA, Nelson and Barghi (1989) suggested that application of 1.23 per cent AFF for 10 minutes results in an effective bond strength similar to HFA applied for 1 minute. On the other hand, etching ceramic surfaces with 37 per cent phosphoric acid was reported to produce a clinically acceptable bond strength comparable with that produced by the application of HFA (Yen *et al.*, 1993; Bourke and Rock, 1999; Larmour *et al.*, 2006).

Silane coupling agents have been reported to enhance bond strength to porcelain surfaces (Newman *et al.*, 1984; Wood *et al.*, 1986; Kao *et al.*, 1988; Winchester, 1991; Bourke and Rock, 1999; Kocadereli *et al.*, 2001). The silane reacts with the silica within the porcelain and the organic groups of the bonding resin, thus forming a bridge between the two materials (Newman *et al.*, 1984; Kern and Thompson, 1994).

The aims of this study were to evaluate the shear bond strength (SBS) of metal and ceramic brackets bonded to two different all-ceramic crowns, IPS Empress 2 and In-Ceram Alumina, to compare the SBS between HFA, phosphoric acid etched, and non-etched all-ceramic surfaces; compare the SBS between IPS Empress 2 and In-Ceram crowns, to investigate the mode of adhesive failure after debond; and evaluate the integrity of the ceramic crowns after debond.

Materials and methods

Ninety-six all-ceramic crowns resembling maxillary first premolars were fabricated utilizing a silicone index. Two types of ceramic crowns were prepared; lucite-based IPS Empress 2 crowns (n = 48) and glass infiltrated In-Ceram alumina crowns veneered with VM7 feldspathic porcelain (n = 48).

The IPS Empress 2 and the In-Ceram ceramics were divided into groups of 12 crowns as follows:

- 1: metal brackets bonded to HFA-etched IPS Empress 2 crowns.
- 2: metal brackets bonded to HFA-etched In-Ceram crowns.
- 3: ceramic brackets bonded to HFA-etched IPS Empress 2 crowns.
- 4: ceramic brackets bonded to HFA-etched In-Ceram crowns.

- 5: metal brackets bonded to phosphoric acid-etched IPS Empress 2 crowns.
- 6: metal brackets bonded to phosphoric acid-etched In-Ceram crowns.
- 7: metal brackets bonded to sandblasted non-etched IPS Empress 2 crowns.
- 8: metal brackets bonded to sandblasted non-etched In-Ceram crowns.

Ceramic crowns were deglazed by aluminium oxide sandblasting with 50 μ m abrasive powder with a microetcher at 80 psi for 2 seconds through a nozzle at a distance of 10 mm and an angle of 45 degrees. After sandblasting, the crowns surfaces were cleaned with water and dried with oil free compressed air.

In the first four groups, the surfaces were etched with 9.6 per cent HFA gel for 2 minutes while in groups 7 and 8, the crowns were etched with 37 per cent phosphoric acid gel for 1 minute. The acid was rinsed away with water and dried with oil free compressed air. In groups 5 and 6, no acid etch was used. This was followed by a silane coupling agent. Tranbond XT primer (3M/Unitek, Monrovia, Bohemia, California, USA) was applied to the etched surfaces in a thin film. Transbond XT adhesive paste was applied to the bracket base (Ominarch metal brackets and Allure Ceramic brackets, 0.022 inch Roth prescription, GAC International Inc., New York, USA) and the bracket was positioned and pressed firmly on the ceramic crowns. Excess adhesive was removed from around the bracket base using a probe and the adhesive was light cured for 40 seconds. The composite resin (Transbond XT) was light cured using a light emitting diode (Ultra-Lite 5 Turbo, Rolence enterprise Inc., Hsin-Chuang City, Taiwan). The light was applied on the interproximal surfaces of the bracket for 10 seconds each. A 1 cm long 0.017×0.025 inch rectangular stainless steel archwire was ligated into the orthodontic bracket slot.

All crowns were cemented with glass ionomer cement (universal glass ionomer cement, Super Dent, Westbury, New York, USA) on dies prepared by clear autopolymerizing polymethyl metahacrylate acrylic resin (PMMA; Acrylic Melliodent, Heraeus Kulzer, GmbH, Ettlingen, Germany). Thereafter, the specimens were embedded in custom-made specimen blocks in clear PMMA. The resin covered the occlusal surface of the all-ceramic crowns with the test surface exposed.

After polymerization, the specimens were transferred to a water bath at 37° C for 24 hours. Subsequently, they were thermocycled from 5 to 55° C and back to 5° C 500 times. The exposure in each bath was 60 seconds and the transfer time between baths 20 seconds.

The specimens were mounted on Universal Testing Machine (Instron 1195, Instron Limited, High Wycombe, Buckinghamshire, UK) with the tensile load applied parallel to the buccal surface of the restoration in a gingivo-occlusal direction. The machine had an upper jaw that was mounted to a movable crosshead and a lower jaw mounted on the base. The crosshead moved at fixed rate of 1 mm per minute at a full scale of 200 Newton (N) until failure occurred. The force required to debond the brackets was recorded in Newton.

After bond failure, the different groups were masked and the bracket bases and ceramic surfaces were examined visually by a single operator (IAAA) to determine the amount of composite resin remaining according to the modified Adhesive Remnant Index (ARI; Årtun and Bergland, 1984; Bishara *et al.*, 1999). According to Bishara *et al.* (1999), the ARI scale ranges from 1 to 5:

- 1. All adhesive remaining on the enamel with the impression of the bracket base.
- 2. More than 90 per cent of the adhesive remaining on the enamel surface.
- 3. Less than 90 per cent but more than 10 per cent of the adhesive remaining on the enamel surface.
- 4. Less than 10 per cent of the adhesive remaining on the enamel surface.
- 5. No adhesive remaining on the enamel surface.

In order to evaluate the type of bond failure at the bracket adhesive interface in each test group, the debonded bracket bases were examined using scanning electron microscopy (SEM; FEI, Quanta 200, Göteborg, Sweden).

Damage to the ceramic surface which may have occurred during shear bond testing was recorded using the Porcelain Fracture Index (PFI; Bourke and Rock, 1999). The index is divided into four scores as follows:

- 0. ceramic surface intact or in the same condition as before the bonding procedure;
- 1. surface damage limited to glaze layer or very superficial ceramic;
- surface damage which features significant loss of ceramic requiring restoration of the defect by composite resin or replacement of the restoration;
- 3. surface damage where the core material has been exposed due to the depth of the cohesive failure.

Method error

Ten randomly selected crowns were re-examined by the same examiner after a period of 1 week, and the kappa test was applied to test intra-examiner reliability. Kappa values were above 92 per cent for the ARI and PFI.

Statistical analysis

The mean and standard deviation (SD) of each group were calculated. Comparison between groups was performed using a univariate general linear model with SBS as the dependent variable and the type of bracket, type of porcelain surface, and surface treatment as fixed variables. Bonferroni *post hoc* multiple comparisons were used. Comparison between the different adhesives and modes of failure was carried out using the chi-square test.

Results

The mean and SD of the SBS of the different groups are shown in Table 1. The highest mean SBS was 120.15 ± 45.05 N which was recorded in group 3 when ceramic brackets were bonded using HFA on the IPS impress ceramic surface, whereas the lowest mean SBS was 57.86 ± 26.19 N which was recorded in group 8 when metal brackets were bonded to In-Ceram ceramic without acid etching. The only factor which significantly affected the SBS was surface treatment (Tables 2 and 3). The SBS of the HFA etched, phosphoric acid etched, and sandblasted non-etched groups

 Table 1
 Means and standard deviation (SD) of the shear bond strength (SBS) (N) of the different surface-treated ceramic crowns.

Group	Description	SBS, mean \pm SD		
1	Metal brackets, HFA, IPS Empress 2	101.70 ± 52.94		
2	Metal brackets, HFA, In-Ceram	106.82 ± 34.83		
3	Ceramic brackets, HFA, IPS Empress 2	120.15 ± 45.05		
4	Ceramic brackets, HFA, In-Ceram	115.18 ± 32.57		
5	Metal brackets, phosphoric acid, IPS Empress 2	110.30 ± 36.97		
6	Metal brackets, phosphoric acid, In-Ceram	87.00 ± 37.11		
7	Metal brackets, sandblasted non-etched IPS Empress 2	59.72 ± 27.33		
8	Metal brackets, sandblasted non-etched In-Ceram	57.86 ± 26.20		

HFA, hydrofluoric acid.

Table 2 F and P values for the effect of the studied variables onshear bond strength.

Variable	F values	P values	
Type of bracket	1.547	0.217	
Type of porcelain	1.203	0.276	
Surface treatment	11.137	***	
Type of bracket × type of porcelain	0.219	0.641	
Type of porcelain × surface treatment	0.947	0.392	

****P* < 0.001.

Table 3Means, standard error (SE) and 95% confidence interval(CI) of shear bond strength (N) of the different variables used inthis study.

Variable	Туре	Mean ± SE	95% CI
Type of bracket	Metal	87.23 + 4.35	78.60–95.87
	Ceramic	100.64 ± 9.82	81.14-120.15
Type of porcelain	IPS-Empress	99.80 ± 7.56	84.78-114.82
•••	In-Ceram	88.08 ± 7.56	73.06-103.09
Surface treatment	Hydrofluoric acid	110.96 ± 5.39	100.25-121.67
	Phosphoric acid	105.35 ± 9.34	86.80-123.90
	Sandblasted-non-etched	65.50 ± 9.10	47.43-83.57

were 110.96 ± 5.39 N, 105.35 ± 9.34 N, and 65.50 ± 9.10 N, respectively. Bonferroni multiple comparisons tests (Table 4) revealed a significant difference in SBS between the HFA-etched groups and the sandblasted non-etched groups ($P \le 0.001$) and between the phosphoric acid-etched and sandblasted non-etched groups (P < 0.001).

The pattern of bond failure using the ARI in the different groups is shown in Tables 5 and 6. SEM of the bracket bases of the different tested groups is shown in Figure 1a–h. There were statistically significant differences in ARI scores between metal and ceramic brackets debonded from

Table 4Mean differences, standard errors, and P values for theshear bond strength (N) of the surface treatment variable usingBonferroni multiple comparisons test.

Groups	Mean difference ± standard error	P value
Hydrofluoric acid and phosphoric acid	12.31 ± 9.34	0.572
Hydrofluoric acid and sandblasted non-etched	52.17 ± 9.09	0.000***
Phosphoric acid and sandblasted non-etched	39.86 ± 10.57	0.001***

*** $P \le 0.001$.

Table 5Adhesive Remnant Index (ARI) scores for the testedgroups (see Table 1).

ARI scores							
Group	1	2	3	4	5		
1	0	0	0	3	9		
2	0	0	0	2	10		
3	5	0	3	0	4		
4	4	0	0	0	8		
5	0	0	0	1	11		
6	0	0	0	0	12		
7	1	0	1	1	9		
8	0		1	1	10		

HFA-etched IPS Empress 2 (groups 1 and 3; P < 0.01) and In-Ceram (groups 2 and 4; P < 0.05). ARI scores recorded for groups 1 and 3 did not differ significantly from those recorded in groups 2 and 4 with a similar bonding protocol.

The results of PFI in the different groups is shown in Tables 7 and 8. The highest incidence of cohesive ceramic fracture (67 per cent of crowns fractured) was observed while attempting to debond metal brackets from HFA-etched In-Ceram group (group 2). IPS Empress 2 crowns showed lower rates of ceramic fracture (groups 1, 3, 5, and 7).

Discussion

In the present study, IPS Empress 2 and In-Ceram crowns were divided into groups containing 12 crowns fabricated by a single operator simulating the maxillary left first premolar. A minimum of 10 specimens is recommended to perform SBS testing (Fox *et al.*, 1994). However, a sample size greater than 10 per group is recommended for bond strength testing of natural teeth where variations in tooth shape exist (Eliades and Brantley, 2000). Maxillary premolar teeth are the teeth most frequently extracted as an integeral part of orthodontic therapy. Therefore, the premolar tooth form was selected to allow clinical simulation and to compare the outcome of the present study with previously reported investigations (Barbosa *et al.*, 1995; Bourke and Rock, 1999; Kocadereli *et al.*, 2001).

In this study, the SBS of HFA-etched crowns were significantly higher than those in the non-etched groups. This is in agreement with the findings of Al Edris *et al.* (1990) where a threefold increase in bond strength was found after the application of HFA to sandblasted ceramic surfaces. However, other authors found similar bond strength between HFA-etched and sandblasted non-etched groups (Zachrisson *et al.*, 1996; Bourke and Rock, 1999; Kocadereli *et al.*, 2001) while others observed higher values with sandblasting than with acid etching (Schmage *et al.*, 2003; Turk *et al.*, 2006; Karan *et al.*, 2007). Karan *et al.* (2007) compared the effect of sandblasting alone with that of sandblasting and HFA etching on three ceramic groups, namely feldspathic, lucite-based ceramics, and lithium disilicate ceramics. They

 Table 6
 Levels of significance for the Adhesive Remnant Index scores for the tested groups (see Table 1).

	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
			**	*				
Group 1			4.4.					
Group 2			**	*				
Group 3	**	**			**	**		*
Group 4	*	*				*		
Group 5			**					
Group 6			**					
Group 7								
Group 8			*					

*P < 0.05, **P < 0.01.



Figure 1 Scanning electron photomicrographs of (a) metal bracket debonded from hydrofluoric acid (HFA)-etched IPS Empress 2 crown; (b) metal bracket debonded from HFA-etched In-Ceram crown; (c) ceramic bracket debonded from HFA-etched IPS Empress 2 crown; (d) ceramic bracket debonded from HFA-etched In-Ceram crown; (e) metal bracket debonded from phosphoric acid-etched IPS Empress 2 crown; (f) metal bracket debonded from phosphoric acid-etched IPS Empress 2 crown; (g) metal bracket debonded from sandblasted non-etched IPS Empress 2 crown; (h) metal bracket debonded from sandblasted non-etched IPS Empress 2 crown; (h) metal bracket debonded from sandblasted non-etched IPS Empress 2 crown; (h) metal bracket debonded from sandblasted non-etched In-Ceram crown.

reported that the SBS of the acid-etched groups was lower than those of the sandblasted non-etched groups.

In this study, it was found that the phosphoric acid-etched groups had similar bond strengths to those etched with HFA. This is in agreement with Nebbe and Stein (1996), Bourke and Rock (1999), Pannes *et al.* (2003) and Larmour *et al.* (2006) but in contrast to Ajlouni *et al.* (2005).

In the present investigation, metal and ceramic brackets had similar SBS values. This is in agreement with the findings of Willems *et al.* (1997).

No significant differences were found with all surface preparation techniques between the IPS Empress 2 and In-Ceram ceramic groups. However, Turk *et al.* (2006) reported that lithium disilicate had a higher SBS than feldspathic porcelain restorations. Moreover, Abu Alhaija and Al-Wahadni (2007) observed significant differences between feldspathic and lithium disilicate ceramic restorations, with a higher mean SBS reported in the feldspathic porcelain group. This may be due to differences in the processing methods and the molecular structure of the two all-ceramic restorations.

In the present study, a high incidence of adhesive bond failure (scores 4 and 5) was observed for all metal bracket groups. In the ceramic bracket groups, mainly cohesive bond failures (score 1) were observed. These findings are similar to those reported by Willems *et al.* (1997).

Table 7Porcelain Fracture Index (PFI) for the tested groups (see
Table 1).

PFI scores						
Group	0	1	2	3		
Group 1	5	4	3	0		
Group 2	2	2	3	5		
Group 3	5	5	2	0		
Group 4	6	2	2	2		
Group 5	10	1	0	1		
Group 6	6	0	0	6		
Group 7	12	0	0	0		
Group 8	10	0	2	0		

It was also observed that neither the type of ceramic materials nor surface conditioning protocol affected the ARI scores within the groups. These results are similar to the findings of Zachrisson *et al.* (1996), Bourke and Rock (1999), and Turk *et al.* (2006) who all reported adhesive type bond failures.

There was a significant difference in ceramic cohesive failure pattern between the different methods of surface preparation. More ceramic fractures were present in the HFA etch IPS Empress 2 and In-Ceram crowns (groups 1–4) compared with those present within sandblasted specimens, whereas phosphoric acid etched and sandblasted IPS Empress 2 and In-Ceram crowns (groups 5–8) were comparable. This finding is in agreement with those of Bourke and Rock (1999). Larmour *et al.* (2006) reported a similar amount of ceramic cohesive failure among both phosphoric and HFA-etched groups, while Karan *et al.* (2007) found that sandblasting lucite-based IPS Empress revealed more ceramic fractures than HFA-etched surfaces.

Although *in vitro* bond strength studies are useful to provide information about new adhesive materials and bonding techniques, *in vitro* bond strength data should be interpreted with caution. A major drawback of *in vitro* bond strength studies is the difficulty in simulating the complex nature of the oral environment. Variations in temperature, stresses, humidity, acidity, and plaque are impossible to reproduce in the laboratory.

Conclusions

- 1. Both metal and ceramic brackets bonded to HFA-etched IPS Empress 2 and In-Ceram crowns resulted in a similar SBS.
- 2. The type of surface treatment was the only factor that significantly affected SBS.
- 3. The pattern of bond failure of metal brackets was at the adhesive–restorative interface, whereas for the ceramic brackets it was at the adhesive–bracket interface.
- 4. The greatest incidence of ceramic fracture after debonding was observed in the HFA-etched IPS Empress 2 and In-Ceram groups.

Table 8 Levels of significance for the Porcelain Fracture Index scores for the tested groups (see Table 1).

	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Group 1						**	**	
Group 2					**		***	*
Group 3							**	
Group 4							*	
Group 5		**						
Group 6	**							*
Group 7	**	***	**	*		**		
Group 8		*				*		

*P < 0.05, **P < 0.01, ***P < 0.001.

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