

# Influence of surface conditioning on ceramic microstructure and bracket adhesion

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**SUMMARY** The objective of this study was to investigate the influence of different conditioning procedures on various ceramic microstructures and bracket adhesion. Ceramic specimens (feldspathic, leucite, leucite-free, and fluorapatite) were mechanically conditioned ( $n = 20$  per ceramic type) with conventional hydrofluoric acid (5 per cent HF; 60/30 seconds), buffered hydrofluoric acid (9.6 per cent BHF; 60/30 seconds), or sandblasting ( $\text{Al}_2\text{O}_3/\text{SiO}_2$  particles). Silane coupling agents were added for chemical conditioning before bracket bonding. Bracket adhesion was calculated with a shear test in a universal testing machine. The bracket-composite-ceramic interface was further evaluated using the adhesive remnant index (ARI). One specimen of each ceramic/conditioning combination was subjected to qualitative electron microscopy investigation. One-way analysis of variance followed by Tukey's honestly significant difference test were applied for inferential statistics.

Conditioning with conventional 5 per cent HF or sandblasting resulted in significantly ( $P < 0.001$ ) higher bond strengths (mean values: 34.11 and 32.86 MPa, respectively) than with 9.6 per cent BHF (mean value: 12.49 MPa). Etching time or sandblasting particles had no statistical ( $P > 0.001$ ) influence on bond strength. Higher ARI scores were found in the conventional 5 per cent HF and sandblasted groups, when compared with the 9.6 per cent BHF group. Microscopic examination of the conditioned ceramic surfaces showed that leucite and leucite-free ceramics differed most with respect to their surface roughness, though without an influence on shear bond strength (SBS;  $P < 0.001$ ). Bracket adhesion was mostly influenced by the conditioning procedure itself. Sandblasted ceramic surfaces showed sufficient conditioning and bracket adhesion; however, the increased bracket adhesion was associated with a risk of ceramic surface damage.

## Introduction

Adequate bonding of brackets to ceramic surfaces is important due to the increasing number of adults undergoing orthodontic treatment (Pine *et al.*, 2001; Ajlouni *et al.*, 2005). The main differences among currently used silicate ceramics in restorative dentistry are in their chemical composition. Feldspathic ceramics, used for veneers, contain feldspar, silicon dioxide, and kaolin. Further development led to leucite and fluorapatite ceramics, containing leucite or fluorapatite crystals in the ceramic matrix, for enhancing cohesive strength and aesthetics. In contrast, leucite-free ceramics have no crystals integrated into the ceramic matrix and show a more homogeneous matrix (Rosenblum and Schulman, 1997; Brantley and Eliades, 2001; Pröbster, 2001; Pospiech, 2004; Barghi *et al.*, 2006).

As any ceramic surface is inert and does not adhere readily to other materials, attempts have been made to alter the ceramic surface for bracket bonding. Adhesive retention to these ceramic surfaces is achieved through a combination of mechanical and chemical retention, i.e. surface roughness and coupling agents (Calamia, 1983; Schaffer *et al.*, 1989; Ajlouni *et al.*, 2005). Surface roughness may be achieved with different procedures such as etching, sandblasting, laser irradiation, and diamond burs (Brantley and Eliades, 2001).

Hydrofluoric acid (HF) etching is a reliable procedure for bonding ceramic restorations, having a dissolving effect on the superficial layers of silicate ceramics (Calamia, 1985; Sorensen *et al.*, 1991; Thurmond *et al.*, 1994; Tylka and Stewart, 1994; Chen *et al.*, 1998; Blatz *et al.*, 2003). HF etching will result in an inhomogeneous surface configuration in leucite ceramics. Leucite crystals are more soluble than the surrounding glass matrix producing microretentive lacunae after etching (Schmid *et al.*, 1992; Blatz *et al.*, 2003; Ozcan and Vallittu, 2003; Saracoglu *et al.*, 2004; Barghi *et al.*, 2006). HF may be harmful to soft tissues in the oral cavity. As a consequence, buffered hydrofluoric acid (BHF) with reduced toxicity was introduced (Kirkpatrick and Burd, 1995; Schiettecatte *et al.*, 2003). However, the recommended application times and acid concentrations differ greatly (Chen *et al.*, 1998; Gillis and Redlich, 1998; Sant'Anna *et al.*, 2002; Saracoglu *et al.*, 2004). Thus, it remains unclear, if etching time influences bracket shear bond strength (SBS).

Zachrisson *et al.* (1996) promoted sandblasting as another mechanical retention procedure. Aluminium oxide particles are blasted onto the ceramic layer at high pressure leaving a microretentive surface. Conditioning depends on particle size, application time, and angle of impact (Zachrisson

*et al.*, 1996; Blatz *et al.*, 2003; Shiu *et al.*, 2007). This method homogeneously abrades the ceramic layers. Furthermore, silica-coated aluminium oxide particles should enhance adhesion tribochemically (Frankenberger *et al.*, 2000; Ozcan and Vallittu, 2003).

Chemical retention is generated by silane coupling agents (Brosh *et al.*, 1997; Cochran *et al.*, 1997; Schmage *et al.*, 2003). Their organic component binds to the silicate groups of the ceramic and their inorganic component to the methacrylate groups of the adhesive material. Previous investigations have shown a significant influence of silane coupling agents on bond strength to ceramic surfaces (Barghi, 2000; Brantley and Eliades, 2001; Blatz *et al.*, 2003).

Other conditioning methods that avoid the intraoral use of HF and sandblasting have been investigated. Diamond burs and lasers have been used for roughening the ceramic surfaces but have a destructive effect by reducing ceramic integrity (Anusavice, 1996; Nebbe and Stein, 1996; Brantley and Eliades, 2001), while laser conditioning has not yet been developed to an acceptable standard (Akova *et al.*, 2005). Phosphoric acid was found to be less successful than HF because of the absence of a dissolving effect on silicate ceramics (Bourke and Rock, 1999; Pannes *et al.*, 2003; Schmage *et al.*, 2003; Ajlouni *et al.*, 2005; Bishara *et al.*, 2005; Türk *et al.*, 2006). A combination of HF and sandblasting was found to be a more time- and material-consuming procedure (Abu Alhaija and Al-Wahadni, 2007).

This *in vitro* study aimed to evaluate the effect of different conditioning procedures on various ceramic microstructures and bracket adhesion. The null hypothesis tested was that different conditioning procedures have no effect on ceramic microstructure and bracket adhesion.

## Materials and methods

Metal- and all-ceramic veneering materials frequently used in prosthodontics were tested in this *in vitro* study (Table 1). Cuboid specimens [5 mm (width) × 5 mm (height) × 25 mm (length)] were fabricated according to the manufacturer's instructions. While metal-ceramic products were sintered onto cast metal-alloy (Porta Geo Ti Wieladent, Lenzing, Austria), all-ceramic products were sintered onto CAD-CAM manufactured zirconium oxide frameworks (Zeno Zr

**Table 1** Ceramic types and manufacturers.

Ceramic type	Trade name	Manufacturer
Leucite	Kiss	Degudent, Hanau, Germany
	Initial MC	GC, Tokyo, Japan
Leucite-free	Zirox	Wieladent, Lenzing, Austria
	Initial Zr	GC
Feldspathic	Vintage	Shofu, Kyoto, Japan
	Akzent	Vita, Bad Säckingen, Germany
Fluorapatite	D.sign	Ivoclar-Vivadent, Schaan, Liechtenstein
	Emax ceram	Ivoclar-Vivadent

Wieladent, Lenzing, Austria) with a ceramic layer thickness of 1 mm. The four types of ceramic were allocated to each of the six conditioning groups shown in Table 2, resulting in 24 subgroups of 40 brackets each.

The HF gels were rinsed with water and air-dried for 10 seconds in the laboratory. Sandblasting was applied for 2 seconds at an air pressure of 2.5 bar (36 psi). The intraoral blaster was seated on a 10 mm spacer perpendicular to the ceramic surfaces. Silane coupling agents were applied to the pre-treated ceramic surfaces for 60 seconds. Light-cure adhesive (Transbond XT 3M Unitek, Monrovia, California, USA) was applied to each silanated ceramic surface. Nine hundred and sixty adhesive-coated upper central incisor brackets (Victory APC, 3M Unitek) were seated and positioned manually on each conditioned surface. Adhesive light polymerization (1000 mW/cm<sup>2</sup>, 420–480 nm, Ortholux LED 3M Unitek) was initiated for 40 seconds. All specimens were stored in isotonic saline solution (NaCl 0.9 per cent Braun, Maria Enzersdorf, Austria) at 37°C for 24 hours after bonding, followed by thermocycling according to the International Organization for Standardization norm (TR 11450; 500 cycles per 5–55°C).

Shear bond testing was performed with a universal testing machine (Z010-TND Zwick, Ulm, Germany) at a crosshead speed of 1 mm/minute. The specimens were seated in the machine and manually fixed at the extension arm. The shearing wedge was positioned at the bracket base (Figure 1). SBS (MPa = Newton/mm<sup>2</sup>) was measured at debonding and recorded automatically.

The adhesive remnant index (ARI; Årtun and Bergland, 1984; Montasser and Drummond, 2009) was used to

**Table 2** Conditioning procedures and manufacturers.

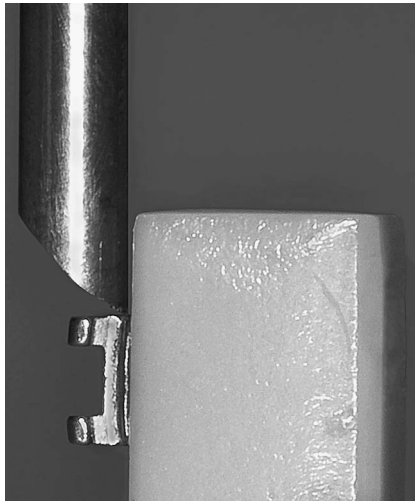
Groups	Conditioning procedure	Trade name	Manufacturer
60" HF 5%	5% HF etching 60 seconds	Ceramics etch*	Vita, Bad Säckingen, Germany
30" HF 5%	5% HF etching 30 seconds	Ceramics etch*	Vita
60" HF 9.6%	9.6% BHF etching 60 seconds	Porcelain-etch**	Ultradent, South Jordan, USA
30" HF 9.6%	9.6% BHF etching 30 seconds	Porcelain-etch**	Ultradent
Al <sub>2</sub> O <sub>3</sub>	50 µm Al <sub>2</sub> O <sub>3</sub> sandblasting	Al <sub>2</sub> O <sub>3</sub> Micron 50*	GAC, Bohemia, New York, USA
SiO <sub>2</sub>	30 µm SiO <sub>2</sub> sandblasting	CoJet™ Sand*	3M Espe, Seefeld, Germany

Silane application for 60 seconds. \*Monobond-S Ivoclar-Vivadent. \*\*Silane Ultradent.

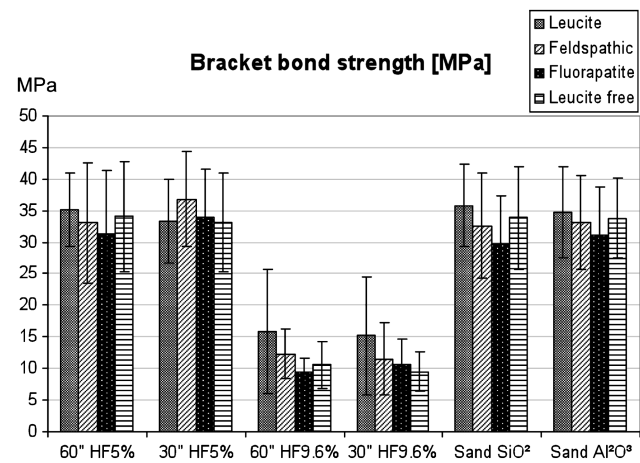
determine the mode of fracture. The ARI and the ceramic fracture rate were evaluated using a stereomicroscope (magnification  $\times 20$ , Mantis FX Vision Engineering, Woking, Surrey, UK). Conditioning combinations were prepared for qualitative evaluation. They were sputter coated for scanning electron microscopy analysis (XL 30-ESEM Philips, Eindhoven, Netherlands).

Data from the shear test were automatically aggregated digitally from a computer coupled to the universal testing

machine. The statistical analyses were run on Microsoft Office Excel 2003 (Microsoft Corporation, Redmond, Washington, USA). Comparisons of the different conditioning procedures were computed by one-way analysis of variance (ANOVA) followed by *post hoc* testing. Tukey's honestly significant difference test was used to determine significance among the different ceramics for each conditioning procedure. ARI and ceramic fractures were evaluated for all conditioning procedures and subjected to chi-square testing. Significance was set at  $P = 0.001$ .



**Figure 1** Photograph of the shear test representing the exact and parallel position of the shearing wedge on the bonded bracket and the ceramic specimen surface.



**Figure 2** Diagram representing the 24 ceramic/conditioning combinations, with mean shear bond strength and standard deviations.

**Table 3** Mean shear bond strengths ( $\bar{X}$ ), minimum (Min), maximum (Max) values, standard deviations (SD) and homogeneous subsets (=the same letters indicate absence of a statistical significance).

Type of ceramic	Groups	$\bar{X}$	SD	Min	Max	Homogeneous subsets
Leucite	60" HF 5%	35.13	5.86	23.68	45.00	B
	30" HF 5%	33.36	6.59	12.88	44.73	B
	60" HF 9.6%	15.92	9.84	4.97	35.65	A
	30" HF 9.6%	15.19	9.37	2.47	35.33	A
	Sand SiO <sub>2</sub>	35.80	6.52	20.97	46.33	B
	Sand Al <sub>2</sub> O <sub>3</sub>	34.72	7.15	17.37	45.10	B
Feldspathic	60" HF 5%	33.08	9.57	13.40	44.62	B
	30" HF 5%	36.80	7.48	20.53	49.61	B
	60" HF 9.6%	12.31	3.88	4.82	23.02	A
	30" HF 9.6%	11.51	5.73	4.65	33.70	A
	Sand SiO <sub>2</sub>	32.57	8.35	15.61	45.23	B
	Sand Al <sub>2</sub> O <sub>3</sub>	33.16	7.37	14.31	43.59	B
Fluorapatite	60" HF 5%	31.36	9.94	8.71	51.01	B
	30" HF 5%	33.97	7.58	13.58	47.53	B
	60" HF 9.6%	9.34	2.35	4.50	15.24	A
	30" HF 9.6%	10.63	4.02	4.75	20.00	A
	Sand SiO <sub>2</sub>	29.74	7.70	11.88	41.19	B
	Sand Al <sub>2</sub> O <sub>3</sub>	31.19	7.49	13.68	42.92	B
Leucite-free	60" HF 5%	34.07	8.68	17.15	47.10	B
	30" HF 5%	33.19	7.87	17.79	51.15	B
	60" HF 9.6%	10.57	3.67	6.87	24.63	A
	30" HF 9.6%	9.53	3.10	3.81	18.32	A
	Sand SiO <sub>2</sub>	33.88	8.12	16.15	48.43	B
	Sand Al <sub>2</sub> O <sub>3</sub>	33.81	6.34	19.35	45.58	B

## Results

Descriptive statistics of SBS (mean, standard deviations, minimum and maximum values including homogeneous subsets) are shown in Table 3 and for each conditioning/ceramic combination in Figure 2.

SBS in the 5 per cent HF-etched groups was higher for the 60 and 30 seconds groups. No significant differences were found either between the groups or between the four ceramic types ( $P > 0.001$ ). SBS in the 9.6 per cent BHF-etched groups was less for the 60 and 30 seconds groups. The difference between these two groups was not statistically significant ( $P > 0.001$ ). Leucite ceramics showed significantly higher bond strength than the other tested ceramics ( $P < 0.001$ ). SBS in the sandblasted groups was higher for the aluminium oxide and silica-coated aluminium oxide groups. There was no significant difference between the groups ( $P > 0.001$ ). Only leucite ceramics in the silica-coated aluminium oxide sandblasting groups showed significantly higher bond strengths than fluorapatite ceramic ( $P < 0.001$ ).

One-way ANOVA and *post hoc* testing showed significantly lower bond strengths for all 9.6 per cent BHF etched groups when compared with the 5 per cent HF etched and sandblasted groups ( $P < 0.001$ ).

ARI scores are shown in Table 4 for each ceramic type. Qualitative analysis of the scanning electron microphotographs showed varying microretentive ceramic surface patterns especially in the HF-etched group. The surface pattern in the leucite ceramic groups are shown in the Figure 3A–3D. The 9.6 per cent BHF etching appeared to cause less leucite dissolution than 5 per cent HF etching (Figure 3A and 3B versus Figure 3C and 3D). The least significant microretentive surface pattern was found in the leucite-free ceramic group (Figure 4A, 4B, and 4C) with a cleaner surface following conditioning. Sandblasting resulted in a homogeneous conditioning effect in all ceramic groups. Sandblasting with aluminium oxide led to a rougher surface than sandblasting with silica-coated aluminium oxide (Figure 3F versus Figure 3E).

## Discussion

Shear testing is a standard procedure for evaluating the SBS of brackets (Oilo, 1993). SBS in the present study was carried out using a standardized protocol as described by Major *et al.* (1995) and Klocke and Kahl-Nieke (2005a,b).

Surface conditioning of all tested ceramics with 5 per cent HF or sandblasting resulted in an adequate SBS for clinical purposes. In contrast, 9.6 per cent BHF showed

**Table 4** Adhesive remnant index (ARI) scores and number of ceramic fractures.

Type of ceramic	ARI scores				Ceramic fractures
	0	1	2	3	
Leucite					
60" HF 5%	—	5	7	20	8
30" HF 5%	—	3	9	21	7
60" HF 9.6%	35	—	3	—	2
30" HF 9.6%	40	—	—	—	—
Sand SiO <sub>2</sub>	—	2	6	23	9
Sand Al <sub>2</sub> O <sub>3</sub>	—	—	7	15	18
Feldspathic					
60" HF 5%	7	6	5	8	14
30" HF 5%	4	4	13	9	10
60" HF 9.6%	40	—	—	—	—
30" HF 9.6%	40	—	—	—	—
Sand SiO <sub>2</sub>	1	3	11	12	13
Sand Al <sub>2</sub> O <sub>3</sub>	—	1	9	12	18
Fluorapatite					
60" HF 5%	4	—	6	8	22
30" HF 5%	3	6	10	13	8
60" HF 9.6%	40	—	—	—	—
30" HF 9.6%	40	—	—	—	—
Sand SiO <sub>2</sub>	1	5	9	11	14
Sand Al <sub>2</sub> O <sub>3</sub>	—	3	7	19	11
Leucite-free					
60" HF 5%	5	5	9	11	10
30" HF 5%	4	6	13	9	8
60" HF 9.6%	40	—	—	—	—
30" HF 9.6%	40	—	—	—	—
Sand SiO <sub>2</sub>	1	1	8	17	14
Sand Al <sub>2</sub> O <sub>3</sub>	—	3	5	17	15

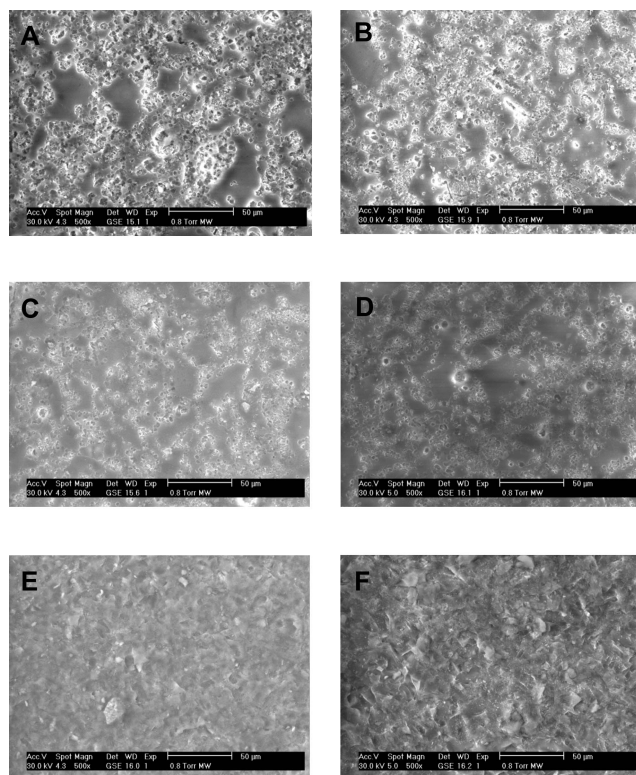
0 = no adhesive left on ceramic surface; 1 = less than half of the adhesive left on ceramic surface; 2 = more than half of the adhesive left on ceramic surface; 3 = all adhesive left on ceramic surface.



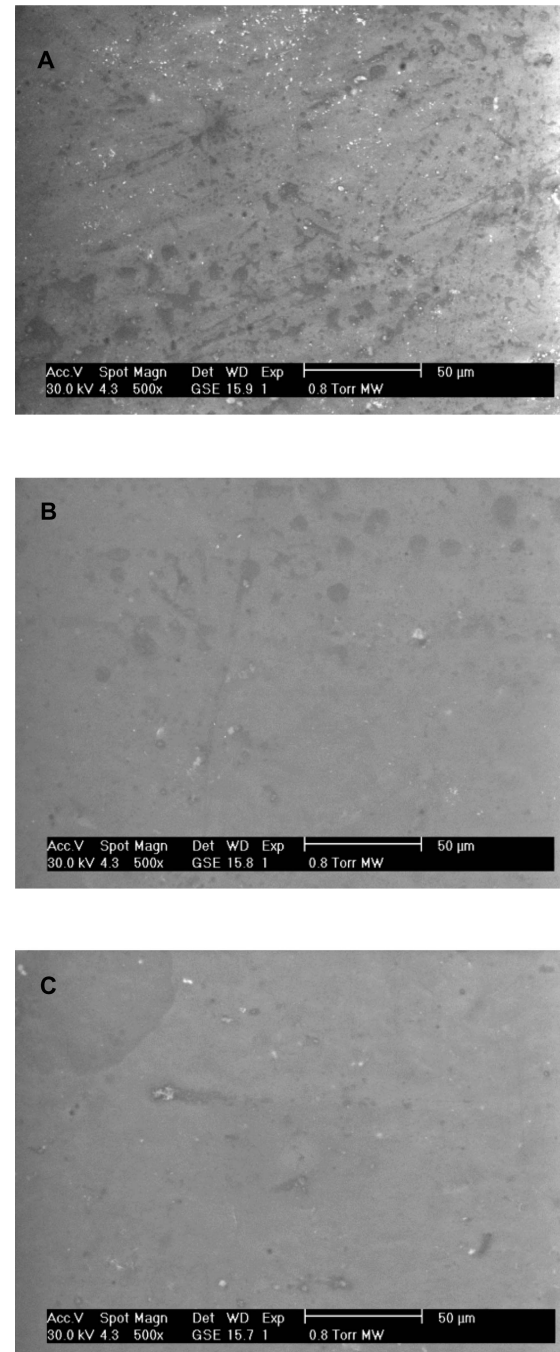
high variability. However, the application times of both HF agents had no effect on SBS. Some authors have reported a difference following sandblasting with aluminium and silica-coated aluminium oxide particles (Vollm, 1989; Meiners *et al.*, 1990). However, the present data did not support these findings. In addition to the high SBS in the 5 per cent HF or sandblasted groups, the ARI scores (2 and 3) indicated strong adhesion between the adhesive and ceramic surfaces, with the majority of the adhesive material left on the ceramic surfaces. In all other instances, the SBS between the ceramic and adhesive exceeded the cohesive strength of the ceramic material and led to ceramic fractures, which should be avoided when debonding (Brantley and Eliades, 2001). The poorer performance in the 9.6 per cent BHF groups compared with all other groups resulted in low SBS and ARI scores (0), i.e. the adhesive remained completely on the bracket mesh base.

Both HF agents produced a microretentive surface, especially in the leucite ceramic groups, by dissolving the leucite crystals in the surrounding glass matrix (Figure 3A–3D). Although the leucite-free ceramics did not show any visible microretentive surface configuration (Figure 4A–4C), high bond strengths were still achieved. It may be speculated that HF etching leads to homogeneous dissolution of the leucite-

free ceramic layers, which is sufficient for bracket adhesion. Sandblasting showed a homogeneous microretentive surface in all the ceramic groups. Surface preparation with aluminium oxide particles seemed to result in a rougher surface than sandblasting with silica-coated particles (Figure 3E–3F). This is consistent with the qualitative findings of Schmage *et al.* (2003).



**Figure 3** Scanning electron photomicrographs showing the six conditioning procedures on leucite ceramic surface (magnification  $\times 500$ ). A: 5 per cent hydrofluoric acid (HF) etching for 60 seconds; B: 5 per cent HF etching for 30 seconds; C: 9.6 per cent buffered hydrofluoric acid (BHF) etching for 60 seconds; D: 9.6 per cent BHF etching for 30 seconds; E: Silica-coated aluminum oxide sandblasting; F: Aluminum oxide sandblasting.



**Figure 4** Scanning electron microphotographs following HF etching on leucite-free ceramic surface (magnification  $\times 500$ ): A: No conditioning; B: 5 per cent hydrofluoric acid (HF) etching for 60 seconds; C: 9.6 per cent buffered hydrofluoric acid (BHF) etching for 60 seconds.

### 5 per cent HF etching

Studies using 5 per cent HF showed SBSs between 12 and 23 MPa (Major *et al.*, 1995; Shahverdi *et al.*, 1998; Bourke and Rock, 1999; Schmage *et al.*, 2003). A short etching time (10–40 seconds) was only investigated in two studies with contradictory outcomes (6/27 MPa; Ozcan and Vallittu, 2003; Saracoglu *et al.*, 2004). Conditioning different ceramics with 5 per cent HF was tested by Ferri *et al.* (2006). In agreement with their results, the present findings are that ceramic type may not have an influence on SBS. Other studies that used the ARI showed similar scores of 3 and ceramic fractures as observed in the present study, but lower SBSs (Major *et al.*, 1995; Bourke and Rock, 1999; Schmage *et al.*, 2003). The high SBS of leucite crystal-free ceramics lacking microretentive surfaces (Figure 4B and 4C) is contrary to reports claiming that SBS is dependent on the presence of leucite crystals in the ceramic surface (Kamada *et al.*, 1998; Estafan *et al.*, 2000; Barghi *et al.*, 2006).

### 9.6 per cent BHF etching

The influence of 9.6 per cent BHF etching has been investigated by several authors (Pameijer *et al.*, 1996; Gillis and Redlich, 1998; Leibrock *et al.*, 1999; Sant'Anna *et al.*, 2002; Ozcan and Vallittu, 2003; Saracoglu *et al.*, 2004; Barghi *et al.*, 2006; Türk *et al.*, 2006; Shiu *et al.*, 2007). In contrast to the present findings, they found that longer etching leads to higher SBS. Conditioning different ceramics with 9.6 per cent BHF was tested by Türk *et al.* (2006). The findings of the present study are in agreement with their results that ceramic type may not have an influence on SBS. Some disparity is found in the literature regarding ARI scores in SBS testing (Cochran *et al.*, 1997; Gillis and Redlich, 1998; Sant'Anna *et al.*, 2002; Larmour *et al.*, 2006).

### Sandblasting

The effect of sandblasting has been tested by numerous investigators (Kao *et al.*, 1988; Smith *et al.*, 1988; Kao and Johnston, 1991; Wolf *et al.*, 1993; Cochran *et al.*, 1997; Gillis and Redlich, 1998; Bourke and Rock, 1999; Sant'Anna *et al.*, 2002; Schmage *et al.*, 2003). SBS ranging from 7 to 41 MPa were found. Sandblasting with silica-coated aluminium oxide showed no significant difference in SBS compared with conventional aluminium oxide sandblasting as also seen in the present study. The same was also observed for the effect of sandblasting different ceramics (Karan *et al.*, 2007). Sandblasted surface preparation led to a high SBS between the ceramic surface and the adhesive material with an ARI score of 3 and ceramic fractures on debonding for all ceramic surface types (Cochran *et al.*, 1997; Gillis and Redlich, 1998; Sant'Anna *et al.*, 2002; Schmage *et al.*, 2003; Türk *et al.*, 2006; Karan *et al.*, 2007).

### Conclusions

The following conclusions can be drawn from this investigation:

- 1 The null hypothesis was rejected. Different conditioning procedures have an effect on ceramic microstructures and bracket adhesion.
- 2 High SBS (29.74–36.80 MPa) were found for all ceramic surfaces when conditioned with 5 per cent HF or sandblasted, indicating a higher risk of ceramic fracture.
- 3 The 9.6 per cent BHF appeared to have a minor conditioning effect, resulting in a lower SBS (9.34–15.92 MPa), but fewer ceramic fractures.
- 4 A short etching time (30 seconds) was as effective as standard etching (60 seconds).
- 5 Silica coating of ceramic surfaces showed no advantage as compared with conventional sandblasting.

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