

Evaluation of alumino-silicate ceramic surface after different surface conditioning methods

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ABSTRACT

Purpose: of this paper was to evaluate changes in alumino-silicate glass ceramic surface structure and the bond strength to composite material after different surface conditioning methods.

Design/methodology/approach: Different conditioning methods of ceramic surface were applied: sandblasting, etching with hydrofluoric acid or both. Grounded ceramic surface served as control group. To evaluate changes of ceramic surface structure CLSM microscopy was used and profilometric analysis of ceramic surface was performed. Shear bond strength of composite material to ceramics was tested.

Findings: The highest bond strength between tested alumino-silicate ceramics and composite material was obtained after sandblasting followed by 5 min 9% HF.

Research limitations/implications: Further research should be conducted to evaluate the influence of surface treatment methods e.g. prolonged hydrofluoric etching time, on mechanical properties of ceramics.

Practical implications: Extending HF etching time of glass-ceramic surface results in increased surface area and allows to obtain high bond strength to composite material.

Originality/value: Results of the study applies to clinical situation as the clinical outcome and survival rate of dental all-ceramic prosthetic restorations depend on reliable bonding to tooth structures.

Keywords: Ceramics; Surface conditioning; Roughness; Shear bond strength

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PROPERTIES

1. Introduction

The all-ceramic dental restoration gain more and more popularity thanks to their aesthetics and biocompatibility. The quality of bonding of dental ceramics to hard dental tissues highly depends on the ceramic surface treatment. The conditioning method of ceramic surface may change its surface structure, influencing the bond strength between ceramic and composite material. The clinical outcome of all-ceramic restorations made of alumino-silicate glass ceramics mostly results from reliable adhesive bonding to hard dental tissues by means of resin-based materials rather than good mechanical properties of the ceramics.

The structure of etched ceramic surface as well as the bond strength between ceramic and composite material depend on many factors like ceramic composition, type and concentration of etchant [1-4]. Hydrofluoric acid is one of the most efficient etchant of alumino-silicate ceramics [5,6]. Ceramics containing more than 15 wt% of silica are classified as alumino-silicate ceramics. Hydrofluoric acid etching together with other adhesive surface treatment methods, air-borne particle abrasion or silanization, result in substantial increase in bond strength between ceramic and resin-based material [5,7,8].

The influence on hydrofluoric acid on alumino-silicate ceramic surface structure depends on etching time and acid concentration [4, 9]. Applying higher concentration of HF for shorter period of time causes selective dissolution of ceramic glassy matrix with untouched crystalline phase. It results in irregular porosities on ceramic surface that enhance adhesive flow. Etching ceramic surface with lower HF concentrations for prolonged period of time resulted in dissolving both glassy and crystalline ceramic matrix. It is reported that excessive crystalline matrix dissolution may lead to reduction of microporosities and affect retentive properties of etched ceramic surface [9,10]. It mostly applies to traditional feldspathic ceramics composing of amorphous glassy matrix made of silicate network with immersed insoluble feldspar and leucite crystals. HF preferentially dissolves leucite crystals [11]. In case of ceramics with higher crystalline phase content, selective dissolution of glassy matrix occurs with crystalline phase intact. Other researchers reported that both, prolonged HF etching of ceramic surface as well as higher concentrations of HF etchant used may result in lower bond strength of ceramic to composite material [3,12] and cause the decrease of flexural strength of etched ceramics [13]. Hydrofluoric acid etching time of ceramic surface reported in the literature vary from 60 seconds to 20 minutes [9,14-16]. Since using hydrofluoric acid in dental practice pose potential health hazard to the patient and to all dental staff,

applying HF higher concentrations or extending the etching time should thoroughly considered.

2. Materials and methods

2.1. Materials

The study used two silicate glass ceramics: Antagon (Elephant Dental) and Carrara (Elephant Dental). Disc-shaped specimens, 5 mm in diameter and 4 mm high, were made of Antagon and Carrara ceramics in layering and press technique respectively, according to manufacturer's instructions. Ceramic surface was ground with 600-grit SiC paper. Samples of each ceramics were randomly divided into 6 study groups (n=13) and one of the following surface conditioning method was applied: 9% hydrofluoric acid etching for 3, 5 or 10 minutes (HF3, HF5, HF10), air-borne particle abrasion with 50 μm Al_2O_3 for 10 seconds at 2.5-bar pressure, followed by hydrofluoric acid etching for 3, 5 or 10 minutes (P+HF3, P+HF5, P+HF10). Ceramic surface as polished served as the control group.

2.2. Surface topography

Images of ceramic surface after application of different conditioning methods were captured using inverted metallurgical microscope (Eclipse MA200, Nikon, Japan) with confocal attachment and changes in ceramic surface topography were observed.

2.3. Profilometric analysis

The surface roughness analysis was performed using inverted metallurgical microscope (Eclipse MA200, Nikon, Japan) with confocal attachment. Profile roughness parameters such as R_a , R_q , R_z , R_y , R_{mr} and R_{v_o} were evaluated, where:

R_a – arithmetic average roughness;

R_q – root mean square roughness;

R_z – average maximum height of the profile (average peak-to-valley height);

R_y – maximum height of the profile;

R_{mr} – bearing ratio;

R_{v_o} – the deepest valley below the centre line; it is an indicator of oil retention or the mechanical behaviour of the surface under high stress (volume measure).

2.4. Shear bond strength

Ceramic samples were invested in PMMA in PVC rings and ceramic surface were treated with one of the method

described above. Afterwards, on ceramic surface silane coupling agent (ESPE Sil/3M ESPE) was applied and allowed to dry for 5 minutes. Then, bonding agent (Adper Single Bond 2/3M ESPE) was brushed into the ceramic surface, air-dried and polymerized with LED polymerizing lamp (Demetron A.2, Kerr, Switzerland). The bonding between ceramic and composite material (Filtek Supreme/3M ESPE) was performed using cylindrical silicone mould, with inner opening of 3 mm in diameter. Composite material was applied in layering technique and polymerized consecutively. Shear bond strength (SBS) was tested after samples' 24-hour storage in 0.9% NaCl. Test was conducted in universal testing machine Z005 (Zwick/Roell) at crosshead speed 2 mm/min, according to ISO/TS 11405 [17].

2.5. Statistical analysis

Within the statistical analysis, generalized linear models with robust standard errors (due to small sample sizes) were fitted, considering both main effect and multifactor solutions. A level of $P < 0.05$ was considered statistically significant. All the statistical procedures were carried out using Stata®/SE release 14.1 (StataCorp LP, College Station, Texas, USA).

3. Results and discussion

3.1. Surface topography analysis

Scale of colour points intensity of images obtained in confocal microscopy indicates the depth of irregularities

on ceramic surface and facilitates surface evaluation. The images of Antagon ceramic surface, presented in Figures 1, 2 and 3, show that HF etching causes substantial change in the ceramic surface structure. Extending etching time from 3 to 5 minutes resulted in increased depth of pits and depressions present on the ceramic surface. Antagon ceramic surface HF etched for 5 minutes showed elevated planes as well as wide depressions with single much deeper pits. The images of Carrara ceramic surface are presented in Figures 4, 5 and 6. In comparison to Antagon, images of Carrara surface is distinguished by more uniform colour dispersion. Dark dots, representing the deepest spots on the surface, are almost homogeneously spread cross the image of the surface.

3.2. Roughness

The results of surface roughness profilometric analysis were presented in tables. For Antagon ceramics (Tab. 1), the highest values of all roughness parameters were obtained after 10-minute HF etching. In case of Carrara ceramics (Tab. 2), sandblasting followed by 10-minute HF etching resulted in the highest values of all R parameters. The lowest R values, for both ceramics, were observed in the control groups. The highest bearing ratio, for both ceramics, was obtained in control groups. Also, extending etching time from 3 to 10 minutes resulted in the increase of R values for grounded surface of Antagon and sandblasted surface of Carrara.

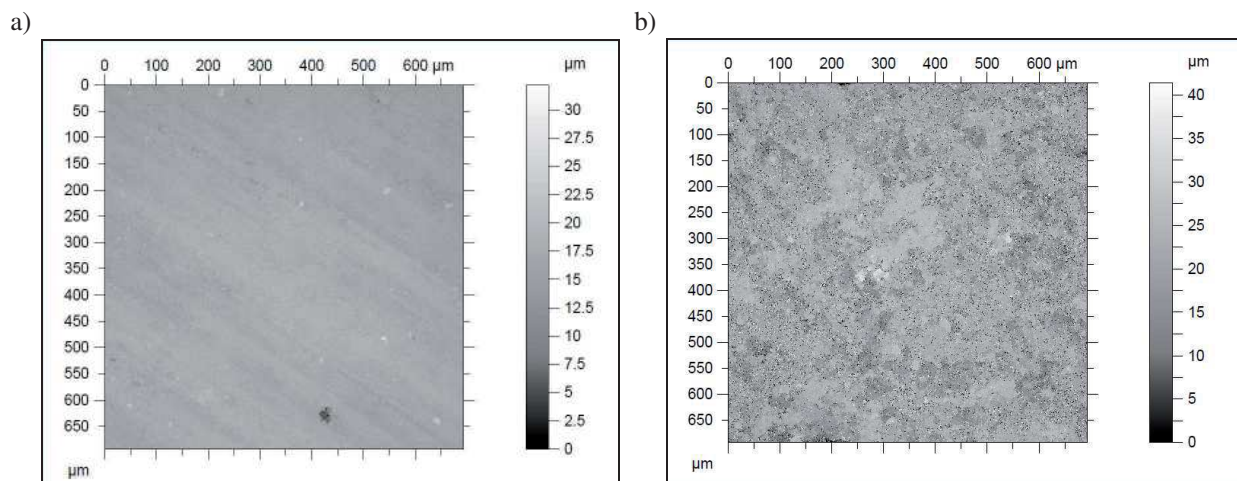


Fig. 1. Antagon ceramics surface: A – control (grounded); B – after 3-min HF etching

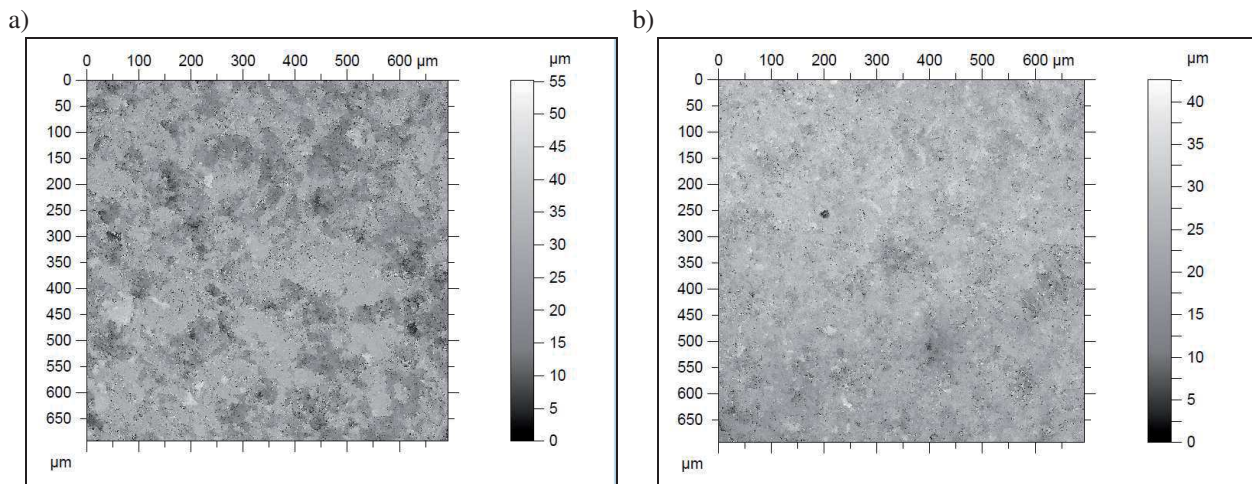


Fig. 2. Antagon ceramics surface after: A – 10-min HF etching; B – sandblasting and 3-min HF etching

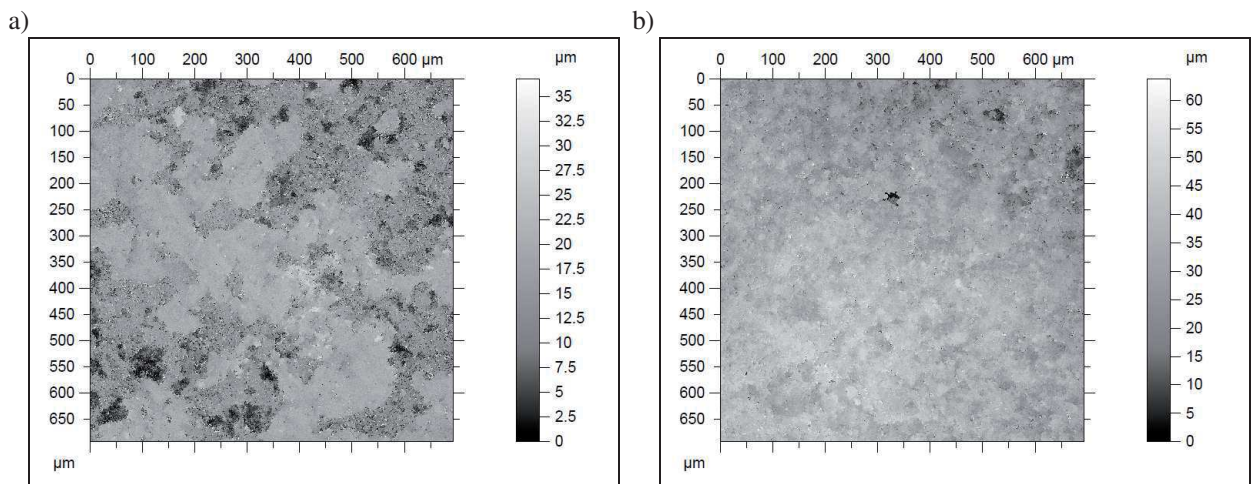


Fig. 3. Antagon ceramics surface after sandblasting and HF etching for: A. 5 min; B – 10 min

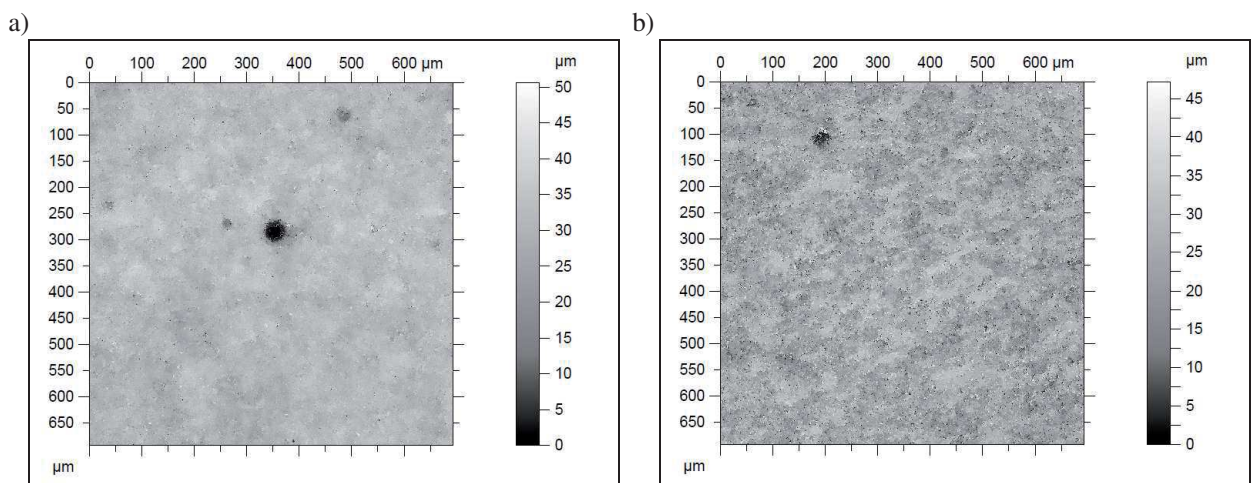


Fig. 4. Carrara ceramics surface: A – grounded (control); B – after 3-min HF etching

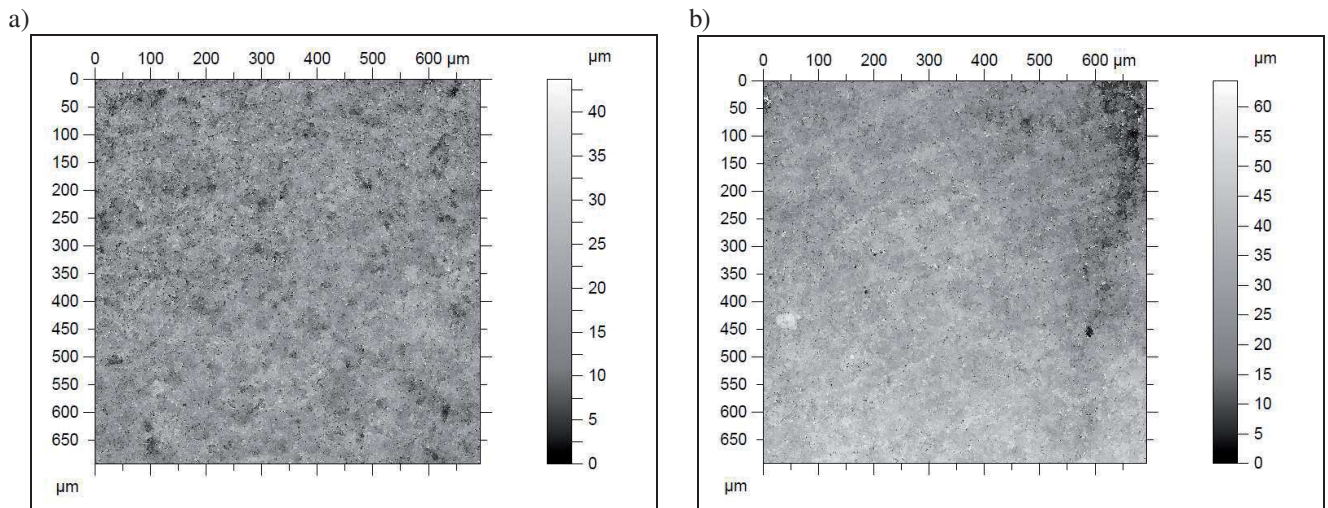


Fig. 5. Carrara ceramics surface after: A – 5-min HF etching; B – sandblasting and 3-min HF etching

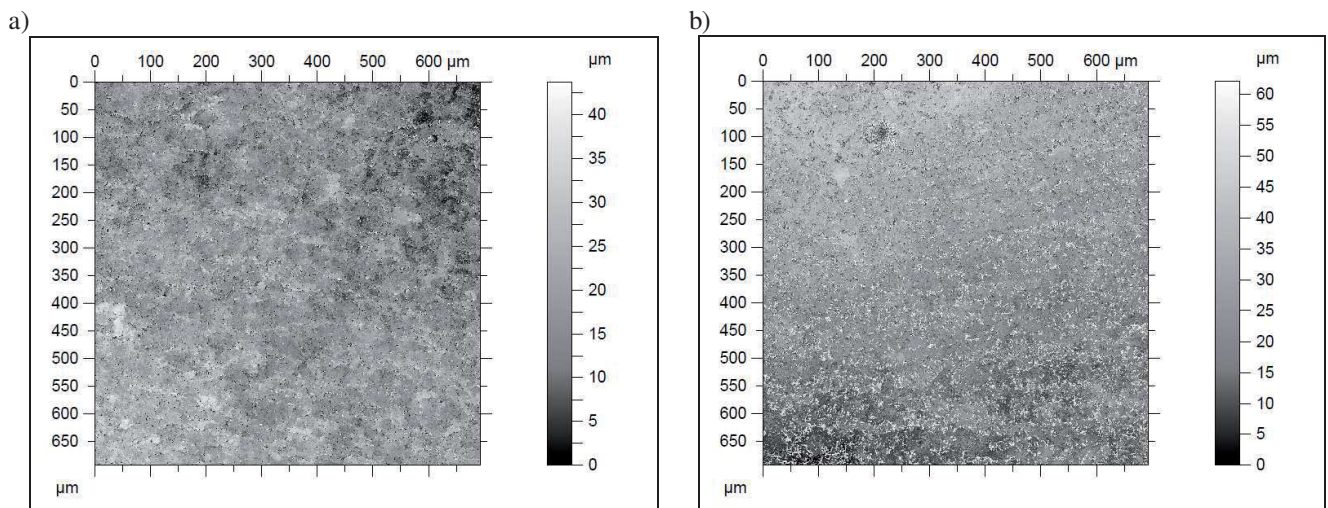


Fig. 6. Carrara ceramics surface after sandblasting and HF etching for: A. 5 min; B – 10 min

Table 1.
Mean values of surface roughness parameters for Antagon ceramics

Surface treatment method	Profile roughness parameter					
	R_a , μm	R_q , μm	R_z , μm	R_y , μm	R_{mr} , %	R_{Vo} , mm^3/mm^2
Control	0.33	0.74	6.24	9.20	76.76	9.58e-006
HF 3	2.81	3.87	26.8	31.00	26.79	0.00484
HF 5	2.30	3.46	28.60	34.50	25.89	0.0117
HF 10	4.08	5.25	33.50	41.40	25.53	0.00935
P+HF 3	2.01	3.01	23.50	28.70	25.91	0.00367
P+HF 5	2.71	3.73	24.60	27.60	25.73	0.00936
P+HF 10	3.42	4.54	28.60	37.40	25.87	0.00386

Table 2.

Mean values of surface roughness parameters for Carrara ceramics

	Surface treatment method	Profile roughness parameter					
		Ra , μm	Rq , μm	Rz , μm	Ry , μm	Rmr , %	R_{Vo} , mm^3/mm^2
Carrara ceramics	control	1.22	1.97	17.90	24.10	66.64	2.17e-005
	HF 3	2.92	3.98	27.90	32.20	28.55	0.00387
	HF 5	3.09	4.28	29.20	35.60	25.95	0.00924
	HF 10	2.97	4.40	36.00	52.90	25.70	0.00494
	P+HF 3	3.51	4.83	35.30	44.80	26.06	0.00825
	P+HF 5	3.17	4.35	28.40	32.20	25.95	0.00706
	P+HF 10	5.96	8.00	45.20	51.70	25.54	0.0196

3.3. Bond strength

The results of shear bond strength of composite material to the Antagon ceramics are presented in Figure 7. The highest mean bond strength values were obtained after ceramic surface was sandblasted, followed by 10-minute HF etching (27.8 MPa). The lowest SBS values were noted after 3-minute HF etching (7.9 MPa).

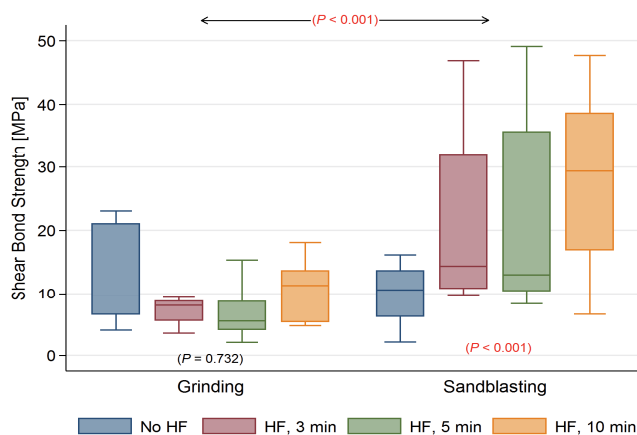


Fig. 7. Statistical analysis of SBS test results [MPa] for Antagon ceramics

The results of shear bond strength of composite material to the Carrara ceramics are presented in Figure 8. The highest mean bond strength values were obtained after ceramic surface was sandblasted, followed by 5-minute HF etching (20.08 MPa). The lowest SBS values were noted after 10-minute HF etching (8.3 MPa).

For both tested ceramics, sandblasting of ceramic surface significantly increased its bond strength to composite material. There was no statistically significant difference in the bond strength of both tested ceramics to composite for various HF etching periods.

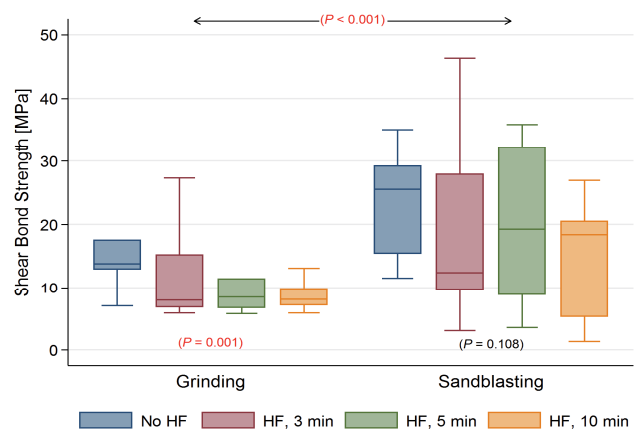


Fig. 8. Statistical analysis of SBS test results [MPa] for Carrara ceramics

3.4. Discussion

As previous study reported [18,19], sandblasting of ceramic surface produce changes in surface morphology of both tested ceramics. SEM images of Antagon and Carrara ceramics showed that the alterations on ceramic surface were more prominent for Antagon ceramics. Similar observations were made by Borges et al. [20] and Akova et al. [21]. Changes in ceramic surface structure were more evident when HF etching was performed. It is reported that increased porosity of ceramic surface positively influence the bond strength of ceramic to resin-based materials [9,22], but also decreases flexural strength of ceramics [23].

In the present study, extending etching time up to 10 minutes resulted in substantial increase of surface area of both tested ceramic. Carrara pressed ceramic after HF etching gains sponge-like or comb-like surface structure [18,19]. Sandblasting of ceramic surface, prior HF etching, positively influenced surface structure, increasing the HF etching effect.

According to Della Bona and van Noort [24], hydrofluoric acid aggressively affects ceramic surface microstructure created during sandblasting. It is also reported that overexposing of ceramic surface to HF etching causes detrimental changes in chemical composition of ceramic surface. Silica disposal of ceramic surface limits the ability to obtain chemical bonding, via silane coupling agents, between ceramic and composite material.

In the present study, profilometric analysis of ceramic surface showed the growth of all R parameters values as the etching time was prolonged up to 5-10 minutes. Addison et al. [13] observed initial increase of feldspathic ceramic surface roughness along with extending the etching time, followed by the decrease of roughness after etching for 3 minutes. Meng et al. [25], after etching leucite ceramic with 5% HF for 30 seconds obtained R_a values below $1\ \mu\text{m}$ and R_y values ranging from 2.5 to $3.5\ \mu\text{m}$, while the highest values of R_a and R_y were noted after sandblasting. Chung and Hwang [26] applied 9.6% HF for 4 minutes on porcelain surface and obtained R_a mean values of $3.33\ \mu\text{m}$. Venturini et al. [27] observed the increase in ceramic surface roughness along with higher HF concentrations applied. According to Ayad et al. [28], values of roughness parameters correlate with ceramic surface treatment method. The values of roughness parameters obtained in the present study did not fully correlate with bond strength test results. For Antagon ceramics, R_a , R_q and R_z values were consistent with the bond strength test results. With the extending etching time of sandblasted ceramic surface, the increase in those roughness parameters values were observed. Similar tendency was observed with the bond strength test results. However, for Carrara ceramics no such tendency was observed. As to R_{vo} , it gives information about fluid retention in surface roughness profile and would indicate the surface potential to retain adhesives or bonding systems. In the present study R_{vo} values were inconsistent with SBS results.

In the present study, prolonged HF application on alumino-silicate ceramic surface, grounded and sandblasted beforehand, resulted in obtaining high shear bond strength values. Statistical analysis of SBS results showed no significant differences between study groups with extended HF etching time from 3 to 10 minutes. However, comparing study groups, where HF etching was preceded by sandblasting, significant growth of SBS values was observed. Moharamzadeh et al. [29], testing fracture toughness of ceramic-composite bond in both dry and wet conditions, observed no significant differences between sandblasting and sandblasting followed by HF etching as long as silane application procedure remained the same. The authors concluded that both ceramic surface treatment

methods are equally effective in obtaining reliable bond strength. Kim et al. [30] proposed the use of universal adhesive system, instead of silane coupling agent, in bonding resin to hydrofluoric acid-etched glass ceramic. Such simplified procedure resulted in improvement of bond strength. The other study [31] showed that either universal adhesive or silane may increase bond strength of self-adhesive cement to HF-etched glass-ceramic, but only in case of universal adhesive application, SBS was significantly reduced after thermocycling.

Altogether, SBS values obtained in the study for both tested ceramics were relatively high. Similar results were reported by Kupiec et al. [7], Thurmond et al. [32], and Lee and Im [33]. They obtained SBS values ranging from 17 MPa to 28 MPa after sandblasting, HF etching and silanization of ceramic surface. Barghi et al. [1] after etching of alumino-silicate ceramic (20-30 wt% leucite content) with 9% HF gel for 1 minute noted approximate SBS values. But with increasing etching time bond strength dropped, yet statistically insignificantly. Stewart et al. [34] after extending HF etching time to 5 minutes achieved bond strength ranging from 16 to 21.5 MPa for different resin-based cements. Other authors like Nagayassu et al. [35] observed that extended etching time of ceramic surface from 2 to 4 minutes resulted in the decrease of ceramic-composite bond strength from 12.74 MPa to 10.99 MPa.

The SBS results of the present study showed no statistical differences between study groups, where different HF etching times were applied. Still, for Antagon ceramics, extended etching time from 3 to 10 minutes resulted in higher mean SBS values, for both grounded and sandblasted surface. In case of Carrara ceramics, the growth of mean SBS values was observed after 5-minute HF etching, followed by the decrease when ceramics was etched for 10 minutes.

Some authors [36] indicate that very short HF etching periods (10-40 seconds) are enough to obtain optimal, retentive ceramic surface topography in SEM images. However, ceramic-composite bond strength values reported are significantly low in comparison to those obtained in the present study.

Moharamzadeh et al. [29] performed assessment of ceramic-composite interface after bond strength test. They observed cohesive and mixed (cohesive/adhesive) fractures within ceramic material in groups where ceramic surface was HF etched or sandblasted and HF etched. They stated that HF etching might have caused the decrease of mechanical strength of ceramic surface, hence the growth of fractures beginning within ceramic material. In the present study the similar observations were made. The prevalence of cohesive fractures in ceramic material was

observed after sandblasting and HF etching of ceramic surface. Some authors [1,37,38] suggests that in case of cohesive fracture within ceramic material, it is hard to explicitly establish the ceramic-composite bond strength value.

Venturini et al. [27] reported that different HF acid concentrations (1%, 3%, 5% or 10%) applied on ceramic surface similarly influenced its flexural strength. HF etching of glass-ceramic, regardless the acid concentration, resulted in substantial decrease of its strength.

Soares et al. [39] stated that sandblasting causes the decrease ceramic mechanical strength and also showed lower initial bond strength. Still, it was the resin-based luting agent layer bonded to ceramic that significantly increased ceramics mechanical strength.

4. Conclusions

Ceramic surface topography differs greatly, depending on method of ceramic manufacturing (layering or press technique). In case of ceramic manufactured in layering technique, surface roughness correspond with the bond strength. For ceramic manufactured in press technique, the higher surface roughness, the lower the bond strength was obtained.

For both ceramics, sandblasting significantly increased bond strength to composite material. Given the results of the study, sandblasting followed by 5-10-minute HF etching seems to be the universal surface treatment method for alumino-silicate ceramic before bonding to composite material.

Still, the further studies should be conducted to evaluate the influence of surface conditioning methods on ceramic mechanical properties (e.g. flexural strength, hardness).

Additional information

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