Lithium Disilicate Glass-Ceramic Surface Appearance after Acid Surface Treatment

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Abstract

Dental ceramics are widely used and studied in dentistry because they are durable, aesthetically appealing and provide excellent biocompatibility. All glass-ceramic surfaces must be etched using hydrofluoric acid (HF) to increase surface roughness determined by roughness average (Ra) before cementation to a tooth surface. This research aimed to analyze the effect of hydrofluoric acid surface treatment concentration on the surface roughness of lithium disilicate glass ceramic. A total of fifteen discs of lithium disilicate glass ceramic were prepared (10mm in diameter and 1mm in thickness). Specimens were divided into 3 groups (n=5). Group A (control) was no treatment, group B was etched by 5% HF for 2 min, and group C was etched by 9.5% HF for 2 min. The etched surfaces were observed by Scanning Electron Microscope (SEM). The measurement of the Ra of the lithium disilicate glass ceramic was determined with surface roughness tester machine. The results showed that the means of Ra (μm) were 0.096±0.009μm, 0.608±0.054μm, and 0.892±0.101μm in group A, B, and C, respectively. The one-way ANOVA showed there was an effect of hydrofluoric acid surface treatment concentration on the surface roughness of the lithium disilicate glass ceramic. The post hoc test showed there was a difference of Ra (μm) among the experimental study groups ($p<0.05$). In conclusion, the concentration of hydrofluoric acid influences Ra of lithium disilicate glass ceramic.

Keywords: Hydrofluoric acid; Surface treatment; Concentration; Surface roughness; Lithium disilicate glass-ceramic.

1. Introduction

Ceramics are inorganic, non-metallic materials which are made by heating raw minerals at high temperatures. All-ceramic restorations are preferable in recent years due to their high esthetics which can mimic the natural teeth appearance, biocompatible and shows good mechanical properties.1

Dental ceramics consist of silicate glass, porcelain, glass-ceramic, or highly crystalline solids. They exhibit chemical, mechanical, physical, and thermal properties that distinguish them from metals, acrylic resins, and resin-based composites2. Lithium disilicate glass ceramic (Li2Si2O5) is one all-ceramic system, currently used in the fabrication of single and multi-unit dental restorations mainly for dental crowns, bridges and veneers because of its color is similar to natural teeth and excellent mechanical properties3.

Hydrofluoric acid (HF) treatment is commonly used on silica-based ceramics to react with, and remove the glassy matrix that contains silica. This treatment leaves the crystalline phase exposed, generating surface roughness4. For ceramic surface treatment, the acid reacts with the glass matrix that contains silica and forms hexafluorosilicate. Selective removal of the glass matrix occurred, and result in crystalline structure exposure. As a result, the surface of the ceramic becomes rough; this is expected for micromechanical retention on the ceramic surface5.

Etching procedure using HF to increase surface roughness and surface free energy must be performed on all glass-ceramic surfaces before cementation. A clinical study showed the importance of bonding where acid etching of glass-ceramic crowns decreases the annual failure risk by about 50%6. In vitro studies reported positive effects of HF etching on the strength of glasses by removing or stabilizing surface defects and on surface topography by increasing the roughness of adhesive bonding7. The current study aimed to analyze the effect of HF surface treatment concentration on the surface roughness of Lithium disilicate glass ceramic.

2. Material and Method

The materials used in this study were: lithium disilicate glass ceramic IPS e.max Press HT (Ivoclar- Vivadent, Schaan, Liechtenstein) and HF solution (ACS, ISO, Reag. Europe). The HF solution was prepared in 5% and 9.5% concentration by adding distilled water.

2.1. Ceramic Preparation

The ceramic blocks (lithium disilicate glass-ceramic) were cut into 10mm in diameter and 1mm in thickness using a low-speed cutter wheel saw under water cooling. The ceramic discs were wet-finished with 800 and 1200 grit silicon carbide paper to remove irregular surface and defects. All ceramic specimens were sonically cleaned with distilled water for 15 min to remove debris.

2.2. Surface Roughness Measurement

In this research, the surface roughness was determined using a surface roughness tester machine (Surf com, 120 A, Japan) and the Ra parameter values were recorded. Ra is the average roughness value of a surface. A lower Ra value results in a smoother surface. The profilometer parameter was set as follows: cutoff length 0.8 mm, transverse length of 0.5 mm. Surface roughness testers were mechanically moved across the surface recording an "image" of the surface roughness across a pre-defined sample length. The roughness tester evaluation results in roughness depth (Rz) and roughness average (Ra) in µm.

2.3. Preparation of Surface treatment

The ceramic specimens were divided into 3 groups (n=15) according to the following ceramic surface treatments:

Group A (control): No treatment was applied to the ceramic surfaces; this group served as a control.

Group B: In this group, the specimens were immersed in a solution of 5% HF for 2 min. After such surface treatment, the ceramic surfaces were rinsed with distilled water for 5 min then air-dried to remove any remnants.

Group C: In this group, the specimens were immersed in a solution of 9.5% HF for 2 min. After such surface treatment, the ceramic surfaces were rinsed with distilled water for 5 min and then air-dried to remove any remnants.

The specimens were stored at 37°C for 24 hours before mechanical testing. As HF offers hazardous effects to health, the ceramic specimens were treated in a laboratory cupboard under ventilation, wearing acid-resistant gloves, coat cover with plastic apron and face shield.

2.4. Scanning electron microscope (SEM) analysis

A qualitative micro-morphological evaluation was performed to one additional specimen from each group. The specimens were sputter-coated with gold and analyzed using an SEM (JEOL/EO, JSM-6510 LA, USA) at 15 kV. Photomicrographs of representative areas for the surface treatments applied on ceramic groups were obtained at 5000 magnification.

2.5. Data Analysis

The data were statistically analyzed using one-way ANOVA and post hoc t-test to determine the significant differences in the ceramic surface roughness values among the different concentrations of HF.

3. Results

Figure 2 showed mean surface roughness of nickel-chromium alloy elevated as the increase of brushing duration. The highest mean of surface roughness (Ra= 0.56µm) was obtained after brushing for 154.5 hours. Measurement of each brushing period showed weight decrease compared to previous period (Fig. 3). Mean weight of nickel-chromium alloy decreased 32% after brushing for 154.5 hours. Figure 3 showed increased surface roughness (Ra) influenced increment of wear volume as indicated by R2 = 0.014 (R = 0.11).

3.1. SEM analysis

The result showed differences among the SEM images of the ceramic surfaces after the different surface treatments. Group A specimens showed smooth and homogeneous surface without any porosity as a result of polishing, and they also had a homogeneous fine grain structure and closed inter-grain space as shown in Figure 1.

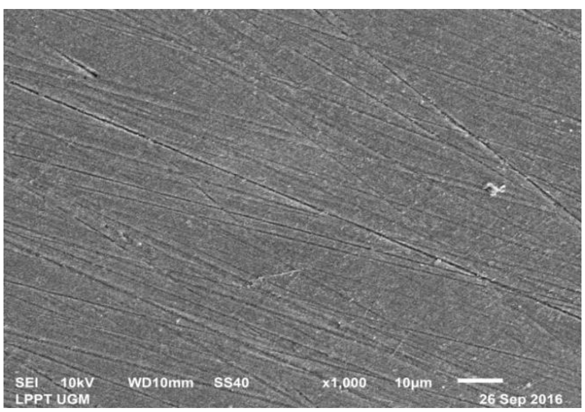

Figure 1. SEM image of group A (control) Group A: smooth surface

Figure 2 showed the SEM images of group B. The etched surface of all the specimens was irregular grain structure with decreased grain size and enlarged inter-grain space compared with that of the control group as a result of the glassy phase.

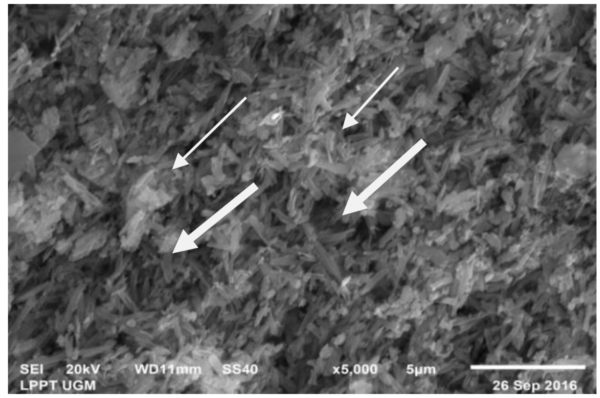

Figure 2. SEM image of group B
grains ⟶ voids

The image of group C which was treated with 9.5% HF and etching time 2 min reveals a further decrease in superficial grains, smaller grains and increased inter-grain space by increased concentrations of acid. The specimens of group C were rougher than that of group B as observed in figure 3.

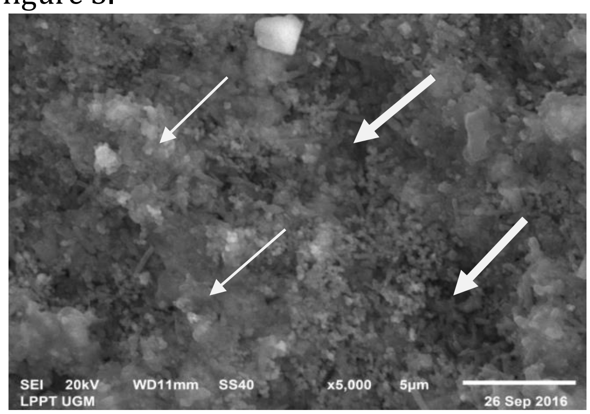

Figure 3. SEM image of group B: etched with 5% HF for 2 min grains ⟶ voids

3.2. Surface roughness analysis

Table 1 showed the mean and standard deviation values of Ra of all the experimental groups. Ra elevated as the acid concentrations of HF in groups B and C increased. The group A specimens showed the lowest surface roughness (0.096±0.009)μm. In the other hand, group C specimens showed the highest surface roughness (0.892±0.101)μm.

Groups	Mean		s.d
Group A (No treat)	0.0960	±	0.00894
Group B (5%)	0.6080	±	0.05404
Group C (9.5%)	0.8920	±	0.10060

Table 1. Mean and Standard Deviation of Ra Results

The differences among the experiment groups on the surface roughness were analyzed by post hoc tests (Table 2). The result proved that there is a significant difference among the experiment groups on the surface roughness (p<0.05).

Groups	Group A	Group B	Group C
Group A	-	51200*	79600*
Group B	-	-	28400*
Group C	-	-	-

*. The mean difference is significant at 0.05 level

Table 2. Results of Post Hoc test (P values) comparing roughness values among treatment groups.

4. Discussion

The purpose of this study was to analyze the influence of different HF etching concentrations on the surface roughness of lithium disilicate glass-ceramic. Based on the results of this research shown in Tables 1 and 2, there were differences in the surface roughness (Ra) of the experimental group which treated with 5% or 9.5% HF. According to Alex8 (2008), the concentrations of 4%–10% hydrofluoric acid are typically used in dental clinic and dental laboratory. These concentration ranges are considered safe for dental applications.

Ceramic etching is a dynamic process and the impact is dependent on substrate constitution, surface topography, acid concentration and etching time9. It is known that HF etching of porcelain provides the necessary surface roughness to mechanical interlocking but over etching could have a weakening effect on the porcelain. Therefore, it is important to know the adequate HF etching concentration for micromechanical retention without weakening the ceramic.

The SEM analysis showed differences in the ceramic surfaces after the different surface treatments of different acid concentrations. The SEM pictures of the ceramic surfaces provided valuable information concerning the result of the topography. The ceramic surface of the control group was smooth and homogeneous (Figure 1). In addition, the surface treatment of specimens using 5% HF (Figure 2) showed porous and irregular structure because of the dissolution of the glass phase. The increased acid concentration of specimens treated with 9.5% HF showed larger and deeper voids and channels (Figure 3). The specimens treated with HF showed morphological changes such as pores and grooves of varied sizes and depths, which are considered to be important to the interlocking of resin to ceramic. This result was in agreement with that of previous studies by Holland et al 10.

Acid etching is the most common in dentistry to improve the bond strength (adhesion strength). Etching porcelain with HF is a gold standard because it creates rough surface required for micromechanical retention with resin composites. Etching increases the surface area by creating micropores penetrating into ceramic to provide durable micromechanical interlocking.

5. Conclusion

Based on the research it can be concluded that the concentration of ceramic surface treatments has a significant influence on the surface roughness of lithium disilicate glass ceramic. Etching with 9.5% concentration of HF of lithium disilicate glass ceramic has provided higher surface roughness compared to etching with 5% concentration of HF.

6. Acknowledgement

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