Key Engineering Materials
ISSN: 1662-9795, Vol. 758, pp 34-38
doi:10.4028/www.scientific.net/KEM.758.34

Submitted: 2017-06-11
Revised: 2017-07-20
Accepted: 2017-09-07
Online: 2017-11-15

Influence of Silane Treatment of All-Ceramic Surfaces (e-Max) on the Cementation with Tooth Dentine

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Keywords: Dental materials, IPS Empress II (e-Max), silane agents, silanization protocols, surface roughness, cementation.

Abstract. The influence of the treatment of the surface of IPS Empress II (e-max) glass-ceramic dental material with different silane agents on the surface roughness of this glass-ceramic was evaluated. IPS Empress II (e-max) cores were treated with five different commercial silane agents used in dentistry nowadays, for several periods of time and were finally air dried. After silanization, the surfaces of the glass-ceramic cores were observed with a light profilometer, a scanning electron microscope (SEM), and an atomic force microscope (AFM). The values of surface roughness (R_a) were also measured with the light profilometer. The results showed an important effect of silane treatment applied for different periods of time, especially for prolonged treatment for 24 hours, on the surface roughness of IPS Empress II (e-max). These results can have an important impact on the deep understanding of the cementation protocols applied in modern dentistry.

Introduction

The evolution of dental-technology is closely bound up with the progress in ceramics. Thus, several high quality dental ceramics and glass-ceramics are currently available, such as Reflex, IPS Empress I, IPS Empress II, Vitablocks, In Ceram, *et cetera*. These materials display outstanding features, in terms of their chemical, mechanical, and aesthetic properties, resulting from their chemical composition, inspired processing, and fine microstructure.

It is a well-known fact in dental practice that the surface treatment of both the ceramic part and the dentine matter of the tooth strongly influence the cementation [1]. More specifically, a literature survey shows that etching with hydrofluoric acid (HF) markedly increases the bond strength. Actually, this etching method is considered to be more effective than other methods, such as sand-blast, or laser, for example [2, 3]. Besides that, it is widely accepted that silane treatment applied on the ceramic surface has a very good effect on bond strength with the natural dentine of the tooth [4]. However, the dentists should precisely follow the protocols suggested by the manufacturers of each type of silane.

Therefore, the aim of this study is to shed light on the influence of the treatment of the surface of IPS Empress II (e-max) glass-ceramic dental material, with five different well-known commercial silane agents, on the surface roughness of this dental glass-ceramic.

Materials and Experimental Procedure

Cores of IPS Empress II (e-max) glass-ceramic were used in cylindrical form (∅ 4 mm, height 6 mm). The surface treatment of the cylinders was done according to the practice followed in dentistry (regular cleaning and mere polishing). For acid etching, HF aqueous solution 10% was used.

As far as the silanization process is concerned, the following silane agents were used: Ceramic Primer (hereafter also abbreviated as CP; by Kuraray), Ceramic Primer Plus (hereafter also abbreviated as CPP, by Kuraray), Porcelain Silane (by BJM Lab.), Silane (by Ultradent), and Monobond-Heliobond (by Ivoclar).

Eighteen cylindrical cores of IPS Empress II (e-max) were used. They were divided into two groups of 9 samples each: the first group was etched with HF solution for 15 s and washed with distilled water for 10 s, and the second group was etched with HF solution for 30 s and washed with distilled water for 10 s.

After the etching with the HF solution, the etched surfaces of the glass-ceramic samples were subjected to silanization treatment by testing all the above silane agents, each one separately. More specifically, for each of the first three silane agents (CP, CPP, and BJM), two protocols of silane treatment were followed: (i) silane treatment for 20 s and then being air dried, and (ii) silane treatment for 24 hours and then being air dried. All specimens were stored in room temperature conditions. The two different times (20 s and 24 h) for silanization were tested because there is not a specific protocol for these three silanes suggested by the manufacturers as far as the silanization time is concerned; thus, there is an open debate in applied dentistry (and literature) whether short silanization time (20 s) or prolonged period of time (24 h) have the best results. On the other hand, the manufacturers for the silanes Ultradent and Heliobond suggest 20 s; thus, only protocol (i) was applied for these two silanes (experiments with 24-h silization would be totally meaningless for the dental practice and, therefore, they were not conducted at all).

Surface roughness measurements (R_a) of the resultant glass-ceramic surfaces were conducted by using a light profilometer (light interferometric measurement microscope for micro-topography measurements, Polytec TMS-1200, resolution 3.65nm). The morphology of these surfaces was also observed under a scanning electron microscope (SEM), equipped with EDS device for element analysis, as well as under an atomic force microscope (AFM).

Results and Discussion

The influence of the surface treatment on the surface morphology of the IPS Empress II (e-max) glass-ceramic is shown in the typical images in Figs. 1a, 1b, and 1c, obtained with the light profilometer for an HF-etched surface for 15 s (Fig. 1a) and after silanization treatment, in 3D view (Fig. 1b) and from horizontal perspective (Fig. 1c). These views (similar views were obtained with all the tested protocols) immediately suggested that silanization had a strong influence on surface roughness. This conclusion was also supported by the SEM observations. More specifically, the typical microstructure of the glass-ceramic IPS Empress II (e-max) was revealed after HF-etching (Fig. 2a), which consists of elongated grains with a high aspect ratio, well-interlocked in the glass matrix, resulting in the outstanding mechanical properties of this superior dental material. In this image, the presence of glassy phase is also obvious. However, silanization had an increasing etching effect (Fig. 2b), apparently in the glassy phase, suggesting that surface roughness should further increase. (Similar conclusions were obtained by the SEM images from all the tested protocols).

The values of R_a measured with the light profilometer for all the tested etching and silanization protocols are summarized in Table 1. Attempts to obtain surface roughness measurements were also conducted with AFM, but they failed in the case of very high roughness values (because roughness was out of the range of AFM). Thus, AFM values merely confirmed the lower R_a values (from the smoother surfaces; a typical AFM image is shown in Fig. 1d) obtained by the light profilometer.

The results of Table 1 support the aforementioned conclusions. More specifically, HF solution etched the surface of the IPS Empress II (e-max). Longer time of etching (30 s) resulted in higher surface roughness than shorter time of etching (15 s). However, this first HF-etching process had a significant influence on the final surface roughness after silanization, since, in all cases, the surfaces treated with HF for 30 s resulted in significantly higher roughness values after silanization with all tested silanes. In some cases, a marked difference was noticed.

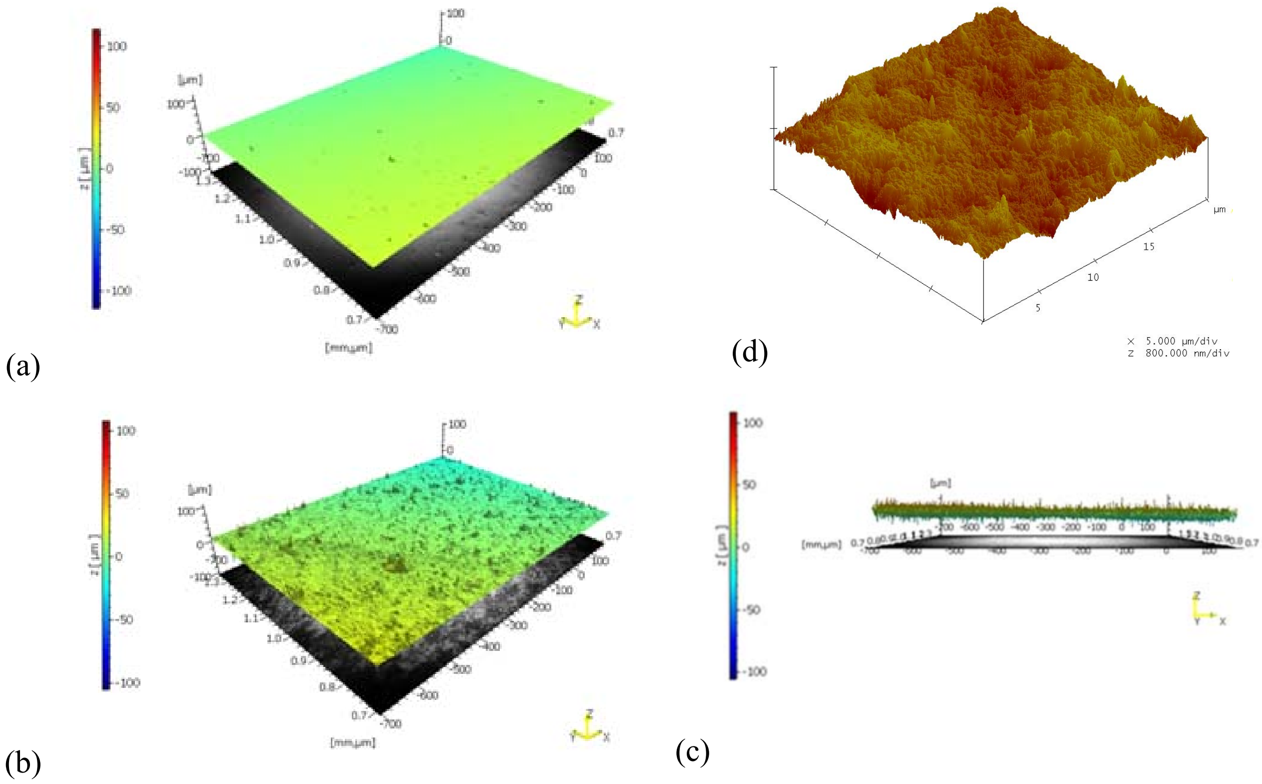
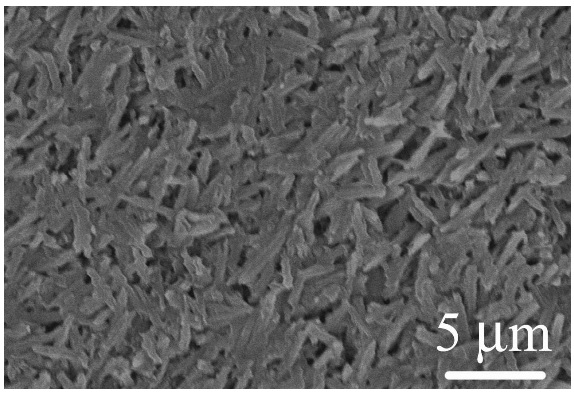
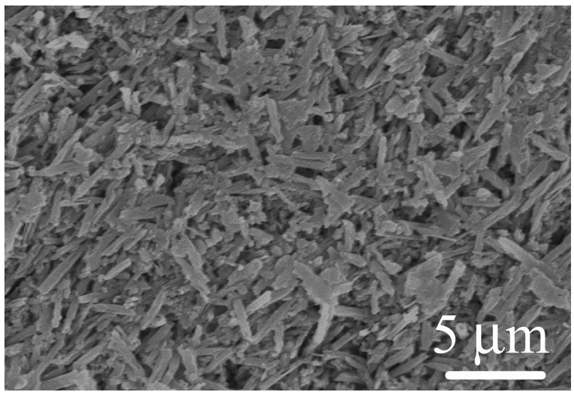


Fig. 1. Typical views of the surface of IPS Empress II (e-max) after HF-etching for 15 s (a), and silanization (b, c, and d). The images (a), (b), and (c) were recorded with the light profilometer. The image (d) was recorded by AFM. In this Figure, (b) and (c) are from the sample HF 15s/CPP 24h, and (d) is from the sample HF 15s/CPP 20s (see Table 1).

(a)


(b)


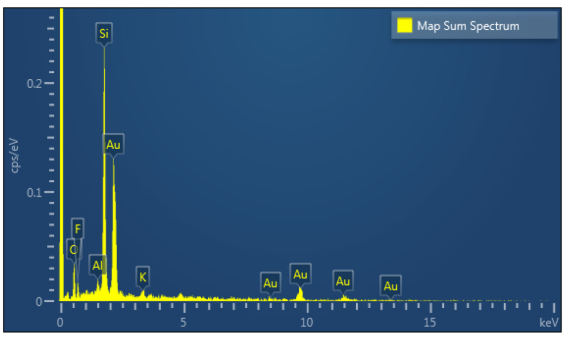
(c)


Fig. 2. Typical SEM images of the surface of IPS Empress II (e-max) after HF-etching (a), and silanization (b), and EDS-spectrum for the elemental analysis of the surface of the glass-ceramic material after silanization (c). In this Figure, (b) and (c) are from the sample HF 30s/BJM 20s (see Table 1).

Table 1. Measurements of surface roughness (R_a) for different etching protocols with HF 10% solution and different silanes applied for different times. (The standard deviation of these values was <10%).

Silane	Time for HF treatment (s)	R_a (μm)	
		no silane treatment	
	15	1.54	
	30	2.07	
		silane treatment 20 s	*silane treatment 24 h*
CP	15	2.22	1.7
	30	2.83	9,0
CPP	15	1.81	3.52
	30	2.23	5.99
BJM	15	10.97	12.25
	30	11.02	13.43
Ultradent	15	7.46	*
	30	13.26	*
Heliobond	15	6.88	*
	30	35.44	*

*: Tests were not conducted (see the materials and experimental procedure section).

There were also differences among the silanes tested. More specifically, the CP and CPP resulted in smoother surfaces whereas much bigger values were obtained with BJM, Ultradent, and Heliobond. The EDS elemental analysis (a typical EDS-spectrum from area elemental analysis is shown Fig. 2c) detected the elements of Si, P, K, Ca, F, and Al on the surface of IPS Empress II (e-max) after silanization. Apparently, these elements are the elements of the bulk glass-ceramic and the silane used. However, the influence of each particular type of silane agent on the surface roughness of IPS Empress II (e-max) is outside the scope of the present paper, as explained in the following lines.

More specifically, the silane agents which are used in dentistry have approximately a similar chemistry (presented in numerous articles in literature). They are generally based on Si-alkoxides. The chemistry of these compounds can result in great improvement of the bond strength between the glass-ceramic material and the natural dentine (actually, they affect interfacial adhesion and wetting behavior of silica-containing glass-ceramics). However, the influence of silane agents on surface roughness of the dental glass-ceramics, which was the main and novel finding in this study, is not a feature that has predominantly been described and interpreted in articles in literature. On the contrary, there is poor documentation on it. Moreover, the EDS analysis conducted in the present work (Fig. 2c), detected the same elements in all five tested silanes. Accordingly, among the silanes which are commercially available on the open market there should be alterations and modifications in the basic composition (e.g. chemical formulae) of the silane agents. Yet, it is obvious that manufacturers never disclose the specific compositions of their products provided for dentists. Therefore, any attempt to interpret the specific influence of each particular type of silane agent tested in this study (reflected in the different R_a values reported in Table 1) is absolutely impossible and it would be purely speculative; thus, it is totally omitted on purpose.

As far as the main finding of this study is concerned, the R_a values from the samples treated with the three silanes CP, CPP, and BJM, clearly showed that the influence of silane treatment on the surface roughness of the glass-ceramic IPS Empress II (e-max) is greatly affected by the different protocols of silane treatment. The results from the treatment with the silanes Ultradent and Heliobond can be witness to the influence of silane treatment on surface roughness (as stressed above, experiments with them for 24-h silanization would be meaningless in dental practice and thus, were omitted). A thorough literature survey revealed only one report [4] which refers to the surface roughness as a result of silanization for a single silane agent. In the present study, not only did we find that all silanes result in an increase in R_a, but also that each of them affects the glass-ceramic surface roughness R_a differently, depending on the different periods of time that this silanization process is applied. More specifically, we found that in the case of CP, CPP and BJM silanes, silane treatment for 24 h led to higher values of R_a compared to the R_a values obtained through shorter silanization periods of time (20 s).

Conclusions

In the course of the cementation process in dental practice, surface roughness is a very important factor in order to achieve a strong and long-term stable interface between the IPS Empress II (e-max) and tooth dentine. This paper reports, for the first time, on the important effect of silane treatment applied for different periods of time, especially for prolonged treatment for 24 hours (for the three silanes CP, CPP, and BJM), on the surface roughness of IPS Empress II (e-max). The results recorded an immediate response of the surface of IPS Empress II (e-max) to silane treatment (proved with all five tested silanes), anticipating an interfacial micro-mechanical stability between the cement and the surface of the glass-ceramic. Therefore, thorough investigation is currently underway to study the influence of these results on the cementation process, and particularly the micromechanical properties of the interface and the bond strength between the IPS Empress II (e-max) and the tooth dentine.

Acknowledgments

The authors are indebted to MSc. Michael Petsios, Dr. Anthi Poulia, and Dr. Antonios Kouloumpis for their kind assistance in SEM and AFM measurements.

References

[1] S.E. Elsaka, Bond strength of novel CAD/CAM restorative materials to self –adhesive resin cement: the effect of surface treatments, J. Adhes. Dent. 16 (2014) 531-540.

[2] A. Della Bona, T.A. Donassollo, F.F. Demarco, A.A. Barrett, J.J. Mecholsky Jr, Characterization and surface treatment effects on topography of a glass-intiltrated alumina/zirconia-reinforced ceramic, Dent. Mater. 23 (2007) 769-775.

[3] Y. Chaiyabutr, S. Mc Gowan, K.M. Phillips, J.C. Kois, R.A. Giordano, The effect of hydrofluoric acid surface treatment and bond strength of a zirconia veneering ceramic, J. Prosthet. Dent. 100 (2008) 194-202.

[4] J.R. Queiros, P. Benetti, M. Ozcan, L.F. de Oliveira, A. Della Bona, F.E. Takahashi, M.A. Bottino, Surface characterization of feldspathic ceramic, using ATR FT-IR and ellipsometry after various silanization protocols, Dent. Mater. 28 (2012) 189-196.