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Surface roughness and gloss of indirect composites etched with acidulated phosphate fluoride solution

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Abstract

Objective. To evaluate the surface characteristics of indirect composites etched with acidulated phosphate fluoride (APF) solution. ***Material and methods.*** APF solution was applied to specimens of six composite materials. The surface morphological change was measured with a confocal laser scanning microscope. Gloss measurement and scanning electron microscope (SEM) observation were also performed. ***Results.*** The APF-treated surface showed various patterns due to the difference in the filler incorporated in each composite. The surface properties could be evaluated precisely from the surface roughness parameters. The arithmetical mean deviation of roughness profile (Ra) parameter of the specimens treated with APF solution was higher than those of the specimens untreated with APF, except New Meta Color Infis (IF). The difference in etching influence of each composite material was shown conclusively in the maximum height of the profile (Rz) parameter. Gradia (GR) and Gradia Forte (GF) were etched three times more deeply than that of IF categorized microfilled composite. The mean width of the profile element (RSm) decreased significantly after APF treatment, except in IF. Gloss was reduced apparently in all materials, indicating that gloss reduction was sensitive to slight surface changes. ***Conclusions.*** Specific filler particles of prosthodontic composite materials were etched by APF application. The surface roughness parameters, such as Rz and RSm, properly described various surface topographic features. Gloss was strongly correlated to surface roughness, as defined by these parameters, and especially to the initial change of surface roughness.

Key Words: *Acidulated phosphate fluoride, composite resins, confocal laser scanning microscopy, surface properties*

Introduction

Application of acidulated phosphate fluoride (APF) solution is effective for caries prevention [1]. Phosphoric acid demineralizes enamel and promotes fluoride uptake, improving the resistance of fluoride incorporated enamel to acid challenge [2]. However, several studies have reported that APF simultaneously attacks inorganic components of dental ceramics [3–6], composites [7–13] and sealants [14]. Irregular surfaces promote the deterioration of gloss [15], staining [16] and plaque accumulation [17], leading to caries and periodontitis. Composite materials have been reinforced with inorganic or organic fillers and improved by reducing the particle size and increasing the filler volume [18]. The improvement in wear resistance and mechanical properties has led to an increase in its use for non-metal prosthetic restorations as well as veneers [19].

A contact stylus instrument is most commonly used for measuring surface roughness. Measurement by this method is limited to the radius of the contact stylus tip, and the risk of surface damage caused by direct contact is high [20]. On the other hand, the non-contact stylus laser method is assumed to provide precise measurement because of its high resolution. Wennerberg reported that typical soft metal biomaterials were best evaluated with the laser approach, whereas hard industrial materials with high slopes within the surface were best evaluated with the stylus instrument [21]. High-quality images and morphological description have been obtained with Atomic Force Microscopy (AFM) [22]; however, AFM measurements are too limited in

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size to allow calculation of some roughness parameters [23].

Surface roughness and gloss of resin composite have been investigated previously. The surface roughness of finished composite is independent of the polishing system, whereas there is interaction of gloss values between composites and the polishing system [24]. Toothbrush abrasion has resulted in significant modifications of the surface roughness and surface gloss among composite materials, probably attributed to filler sizes [25].

Surface roughness of composite etched by APF solution has been examined by contact stylus method and the microfilled material surface was found to be insensitive to APF solution in comparison with a macroinorganic-filled material surface [12,13].

In this study, the influence of APF solution on six indirect composites, including those with recently developed fillers, was evaluated. The aim was to analyze the surface topography of etched-indirect composite by the confocal laser scanning method and quantify the surface property using the surface roughness parameter. Change of surface gloss and the relationship between surface roughness and surface gloss were also examined.

Material and methods

Materials

The materials used in this study are summarized in Table I. Seven indirect composite materials and one machinable ceramic material as the control were examined. New Meta Color Infis (IF) and Ceramage (CE) contained prepolymerized fillers. Pearleste (PE) and Estenia C&B (ES) contained inorganic fillers. Gradia (GR) and Gradia Forte (GF) contained both inorganic and prepolymerized fillers. Froden A (Sunstar, Osaka, Japan), containing 2 g NaF and 1.73 g phosphoric acid in 100 mL solution (pH 3.2 to 3.8), was used as the APF solution.

Specimen preparation

Composite materials were packed into a stainless-steel mold (18 mm × 25 mm × 2 mm), covered with a glass plate, and placed in the center of the light curing apparatus and each side of the specimens was irradiated. The procedures of polymerization are presented in Table II. The polymerizing unit and irradiation time were in accordance with the manufacturers' instructions for each composite material. The sufficient light intensity for the curing unit was checked beforehand using a spectroradiometer (USR-40D; Ushio Inc., Tokyo, Japan). In PE, ES, and GR, heat curing was performed after light curing. ProCAD blocks (CAD) were sectioned to 2 mm thick specimens with a precision machine

Table I. Materials used.

Material	Product name (abbreviation)	Manufacturer	Type/shade	Lot no.	Filler content (wt%)	Filler information
Indirect resin composite	New Meta Color Infis (IF)	Sun Medical Co. Ltd., Moriyama, Japan	Insisal/58	LR1	70	Prepolymerized silica composite
	Pearleste (PE)	Tokuyama Dental Corp., Tokyo, Japan	Ename I/E1	103Y6	82	Spherical silica-zirconia (0.4 μm), spherical silica-titania (0.08 μm)
	Ceramage (CE)	Shofu Inc., Kyoto, Japan	Insisal/58	110608	73	Spherical progressive fine-structured filler
	Estenia C&B (ES)	Kuraray Medical Inc., Tokyo, Japan	Ename I/ E12281	0018AA	92	Splinterd alumino-silicate glass (average 1.5 μm), ultrafine alumina (average 0.02 μm)
	Gradia (GR)	GC Corp., Tokyo, Japan	Ename I/E2	0510261	75 (54+21)*	Splintered glass (<2.0 μm), prepolymerized silica composite
	Gradia Forte (GF)	GC Corp.	Enamel/E1	0511011	76 (73+3)*	Splintered glass (average 1.0 μm), prepolymerized silica composite
Ceramics	ProCAD Blocks (CAD)	Ivoclar Vivadent AG, Schaan, Liechtenstein	I12/E100	JM0441	–	–

*Inorganic filler+prepolymerized filler.

Table II. Polymerization procedures.

Product name	Light curing unit (manufacturer), time	Heat curing unit (manufacturer), temperature, time
IF	α-Light II (J. Morita Corp.,Tokyo, Japan), 1.5 min	–
PE	Pearlcure Light (Tokuyama Dental), 2 min	Pearlcure Heat (Tokuyama Dental), 100°C, 15 min
CE	Solidilite (Shofu), 3 min	–
ES	α-Light II (J. Morita), 5 min	KL-310 (Kuraray Medical), 110°C, 15 min
GR	Labolight LV-II (GC), 3 min	–
GF	Labolight LV-II (GC), 3 min	Petit Oven PO-I (GC), 110°C, 15 min

(Isomet; Buehler Ltd., Lake Bluff, Ill., USA). All test specimens were ground under running water with a series of silicon-carbide papers (#800-2000, WetorDry Tri-M-ite; 3M Corp., St. Paul, Minn., USA) and further polished with wet felt (Texmet, Buehler Ltd.) and a series of diamond suspensions (3, 1, 0.25 μm, MetaDi monocrystalline diamond suspension; Buehler Ltd.). The reduced surface was approximately 500 μm. Specimens were cleaned ultrasonically to remove any remaining polishing debris. Ten specimens were prepared for each material and stored in distilled water at 37°C for 24 h.

APF treatment

APF treatment was performed as described previously [12,13,26]. Briefly, after half the surface was coated with Protect Varnish (Kuraray Medical, Inc., Tokyo, Japan), to protect from the influence of APF application, the entire surface was treated with an APF solution. Ceramic materials were divided into two groups, and one group was coated with Protect Varnish. The APF was applied for 4 min and washing and drying were repeated eight times for each specimen. The varnish film on the untreated side was then carefully removed and all specimens were cleaned ultrasonically for 5 min in distilled water.

Measurement of gloss

Gloss was measured using a gloss meter (GM-26D; Murakami Color Research Laboratory, Tokyo, Japan) with an incident angle of 60°.

Confocal laser scanning measurement

The surface topography was analyzed using a scanning laser microscope (1LM21W; Lasertec, Yokohama, Japan). To evaluate surface roughness, the following parameters were calculated by measuring the two-dimensional surface profile using image analysis and measurement software SALT Ver. 3.60A (Mitani Corp., Tokyo, Japan) [27,28]: arithmetical mean deviation of roughness profile (Ra), root mean square value of roughness profile (Rq), maximum height of the profile (Rz), core roughness depth (Rk), reduced peak height (Rpk), reduced valley depth (Rvk), mean width of the profile element (RSm), and skewness of height distribution (Rsk). Ra, Rq, Rz, and Rsk are amplitude parameters and RSm is a spacing parameter. These are based on the ISO 4287:1997 [27]. Rz, the sum of height of the largest profile peak height and the largest profile valley depth within a sampling length, provided better indication of the actual depth of surface irregularities. Rsk is the quotient of mean cube value of the ordinate values and indicates height character. A surface with predominantly deep valleys tends to have a negative skew, whereas a surface comprised predominantly of peaks has positive skew. Rpk, Rk, and Rvk are derived from the bearing ratio curve based on the ISO 13565: 1996 standards [28]. Rk is the core roughness part with the predominant peaks and valleys removed. Rpk is the peak height above the core part and Rvk the valley depth below the core part [21].

Scanning electron microscopy

Specimens were sputtered with osmium and observed through a scanning electron microscope (SEM; S-4300; Hitachi High-Technologies Co. Ltd., Tokyo, Japan) operated at 15 kV. The specimens of CAD half-coated with Protect Varnish were prepared for SEM observation.

Statistical analysis

The Mann-Whitney U-test (SPSS 14.0J for Windows; SPSS Japan, Inc., Tokyo, Japan) was used to compare the surface roughness parameters between no treatment and treatment with APF. Steel-Dwass multiple comparison (Kyplot 4.0; KyensLab, Inc., Tokyo, Japan) [29] was performed to assess the difference in the surface roughness parameters among the materials treated with APF. The differences were considered to be statistically significant when $p < 0.05$. The correlation between the gloss value and the surface roughness parameters was investigated using the median of each group data by Spearman's rank correlation coefficient (SPSS 14.0J) with the level of statistical significance set as $p < 0.01$.

Results

Table III gives the results of the confocal laser scanning measurement and statistical analysis. The values of Ra were small (0.02 ~0.08 μm) compared to those of other parameters. However, specimens treated with APF solution displayed higher values for Ra than specimens not treated with APF solution, except IF. The values of Rq were increased slightly (0.03 ~0.11 μm) compared to those of Ra, so that there was a significant difference among the materials treated with APF solution. The statistical category of Rq after APF treatment could be described as IF = PE = CE < ES = GR < GF. The value of Rz changed significantly after APF treatment in all composites. The statistical category of Rz after APF treatment could be described as IF < PE = CE = ES < GR = GF. The difference in the value of Rk among materials was small. The values of Rpk and Rvk were largest in GR and GF. The values of RSm decreased greatly after APF treatment, except in IF. The values of ES and GF after APF treatment were relatively small and divided into statistical categories "d" and "e", respectively. The value of Rsk in all tested composite was positive. The parameters of CAD revealed great roughness.

Table IV gives the results of the gloss measurement. Apparent reduction of surface gloss was demonstrated with APF treatment. There was a slight reduction in gloss of IF and GR, while the gloss of CE and ES was markedly decreased. Gloss of CAD disappeared. Correlations between gloss

Table III. Mean (SD) surface roughness.

	Ra							Rq						
Product name	No treatment	APF	Statistical category					No treatment	APF	Statistical category				
IF	0.04 (0.01)	0.04 (0.01)	a					0.05 (0.01)	0.05 (0.01)	a				
PE	0.03 (0.00)	0.05 (0.01)*	a					0.04 (0.00)	0.07 (0.02)*	a				
CE	0.02 (0.00)	0.05 (0.01)*	a	b				0.03 (0.00)	0.07 (0.01)*	a				
ES	0.02 (0.00)	0.07 (0.00)*			c			0.03 (0.00)	0.09 (0.00)*		b			
GR	0.02 (0.00)	0.06 (0.01)*		b	c			0.03 (0.00)	0.09 (0.01)*		b			
GF	0.02 (0.00)	0.08 (0.01)*				d		0.03 (0.00)	0.11 (0.01)*			c		
CAD	0.03 (0.01)	0.11 (0.01)*					e	0.04 (0.01)	0.14 (0.01)*					d
	Rz							Rk						
Product name	No treatment	APF	Statistical category					No treatment	APF	Statistical category				
IF	0.30 (0.09)	0.45 (0.13)*	a					0.11 (0.04)	0.13 (0.04)	a				
PE	0.36 (0.04)	1.09 (0.22)*		b				0.09 (0.01)	0.19 (0.11)*	a	b			
CE	0.32 (0.06)	0.97 (0.12)*		b				0.08 (0.01)	0.17 (0.02)*	a				
ES	0.28 (0.03)	1.09 (0.05)*		b				0.07 (0.01)	0.22 (0.02)*		b			
GR	0.37 (0.03)	1.52 (0.05)*			c			0.08 (0.01)	0.16 (0.03)*	a				
GF	0.45 (0.07)	1.57 (0.09)*			c			0.08 (0.01)	0.23 (0.02)*		b			
CAD	0.41 (0.03)	1.81 (0.09)*				d		0.09 (0.01)	0.40 (0.20)*			c		
	Rpk							Rvk						
Product name	No treatment	APF	Statistical category					No treatment	APF	Statistical category				
IF	0.04 (0.01)	0.04 (0.01)	a					0.03 (0.01)	0.03 (0.01)	a				
PE	0.04 (0.00)	0.09 (0.02)*		b	c			0.02 (0.01)	0.06 (0.02)*	a	b			
CE	0.03 (0.01)	0.08 (0.01)*		b				0.03 (0.01)	0.05 (0.01)*	a				
ES	0.03 (0.00)	0.11 (0.01)*			c			0.03 (0.00)	0.09 (0.01)*		b			
GR	0.03 (0.01)	0.14 (0.02)*				d		0.03 (0.00)	0.14 (0.01)*			c		
GF	0.03 (0.00)	0.16 (0.01)*				d		0.03 (0.00)	0.14 (0.01)*			c		
CAD	0.03 (0.00)	0.16 (0.02)*				d		0.03 (0.00)	0.17 (0.02)*					d
	Rsm							Rsk						
Product name	No treatment	APF	Statistical category					No treatment	APF	Statistical category				
IF	27.92 (6.41)	22.32 (5.77)	a					0.96 (0.45)	0.84 (0.27)	a				
PE	17.38 (1.85)	7.52 (3.12)*		b	c	d		1.16 (0.20)	0.54 (0.24)*	a	b			
CE	23.75 (5.32)	7.89 (1.37)*		b				1.15 (0.29)	0.86 (0.25)*	a				
ES	26.37 (2.24)	5.28 (0.17)*				d		0.77 (0.33)	0.58 (0.11)	a	b			
GR	26.16 (3.25)	6.47 (0.31)*			c			0.87 (0.25)	0.24 (0.36)*		b	c		
GF	26.50 (2.76)	4.57 (0.14)*					e	0.66 (0.42)	0.34 (0.22)*		b	c		
CAD	23.16 (2.74)	6.68 (0.60)*		b	c			0.90 (0.33)	−0.05 (0.24)*			c		

Asterisk (*) indicates significant difference ($p < 0.05$) between no treatment and APF treatment.
Identical letters in statistical category indicate no significantly difference ($p > 0.05$) among APF treated materials.

Table IV. Gloss measurement (mean (SD)).

Product name	No treatment	APF	Statistical category			
IF	82.4 (1.2)	57.4 (1.9)*	a			
PE	76.2 (3.2)	33.6 (3.5)*			c	d
CE	90.4 (0.2)	31.2 (6.0)*			c	d
ES	96.5 (0.9)	31.4 (0.8)*				d
GR	87.1 (0.4)	49.0 (0.6)*		b		
GF	83.4 (1.8)	32.8 (0.8)*			c	
CAD	78.7 (2.9)	4.7 (0.8)*				e

Asterisk (*) indicates significant difference ($p < 0.05$) between no treatment and APF treatment in each composite with the Mann-Whitney U-test.
Identical letters indicate that the values are not significantly different ($p > 0.05$) among the APF treated materials with the Steel-Dwass test.

and the surface roughness parameters are given in Table V. A highly significant negative correlation was observed for Ra, Rq, Rz, Rk, Rpk and Rvk, while a significant positive correlation was observed for RSm and Rsk.

Figures 1 and 2 show the representative border area in SEM images of the tested materials. Irregular surface topography was observed after APF treatment and the surface features differed among materials. No apparent change in IF and PE was observed (Figure 1a, b); however, a slightly etched surface appeared at high magnification (Figure 2a, b). Spherical filler particles of CE and splintered fillers of ES were also etched (Figure 1c, d). Splintered fillers of GR and GF were etched severely (Figure 1e, f). The image of CAD showed excessive surface degradation (Figure 3).

Discussion

The surface profile of etched-composite material was investigated by confocal scanning microscopy. APF treatment was performed under the same conditions of a previous study employing the contact stylus method [26]. The study reported that the values of Ra of GR and IF changed from 0.105 μm to 0.121 μm and from 0.151 μm to 0.163 μm, respectively. GR as a macro-filled material exhibited significant differences between no treatment and treatment with APF. IF as a microfilled material exhibited no statistically significant difference. In the present study, the Ra value of GR changed from 0.02 μm to 0.06 μm, while that of IF remained unchanged from 0.04 μm. The Ra values before APF treatment were small compared to the previous study, because polishing of the specimens was performed effectively with different methods. However, our statistical results were consistent with that report.

Table V. Spearman's correlation between gloss value and surface roughness parameter.

Surface roughness parameter	Correlation coefficient	*p*-value
Ra	−0.923	<0.000
Rq	−0.917	<0.000
Rz	−0.812	<0.000
Rk	−0.938	<0.000
Rpk	−0.866	<0.000
Rvk	−0.841	<0.000
RSm	0.802	0.001
Rsk	0.722	0.004

PE material uses 0.4 μm and 0.08 μm spherical particles as filler. Advantages of this material include easy polishing and specific wear characteristics. The filler-matrix interface may be weak and the filler may readily detach. The surface of this material after APF treatment showed numerous pits; without APF treatment the surface also showed some pits under the polishing procedure. Owing to the small size of the filler, the values of the Ra and Rq parameters after APF treatment were statistically similar to those of IF. The value of RSm decreased significantly.

Surfaces of CE and ES after APF treatment showed concave patterns. The values of the Rz parameter in PE, CE, and ES were about 1 μm. Although there was no significant difference in the Rz parameter between CE and ES, those of the Ra and Rq parameters of CE were smaller than those of ES. This may be due to the low content of inorganic filler of CE compared to ES. The value of RSm of ES after APF treatment decreased markedly.

The characteristic deep concavities of GR and GF were clearly indicated by the Rz parameters. The Rz values of GR and GF were three times greater than that of IF. The values of Rpk and Rvk were also large. The formation of voids could be due to the low acid resistance of the filler components of GR and GF. Comparison of these two materials revealed no differences between the parameters such as Rz, Rpk, and Rvk. However, the Ra, Rq, and RSm parameters were significantly increased in GF, possibly because the proportion of inorganic filler of GF is higher, and that of prepolymerized silica composite lower, compared to GR. The SEM image of CAD revealed excessive surface degradation, porosity, and interconnecting fissures. The height parameters of CAD indicated great roughness.

The gloss was considerably affected by the surface structural changes in the tested materials after APF treatment. The roughness parameters showed a high correlation with gloss values. Kakaboura reported that gloss values were directly linked to surface roughness average (Sa) values obtained by AFM but not to Ra obtained by 2-D profilometry [24]; however, a high correlation coefficient ($r = -0.923$) was found between gloss and Ra values in this study. Although only the Rz parameter indicated a surface change in IF, the deterioration in gloss was apparent, suggesting that gloss responds sensitively to the initial change in surface roughness. IF and GR demonstrated minor changes in gloss because the

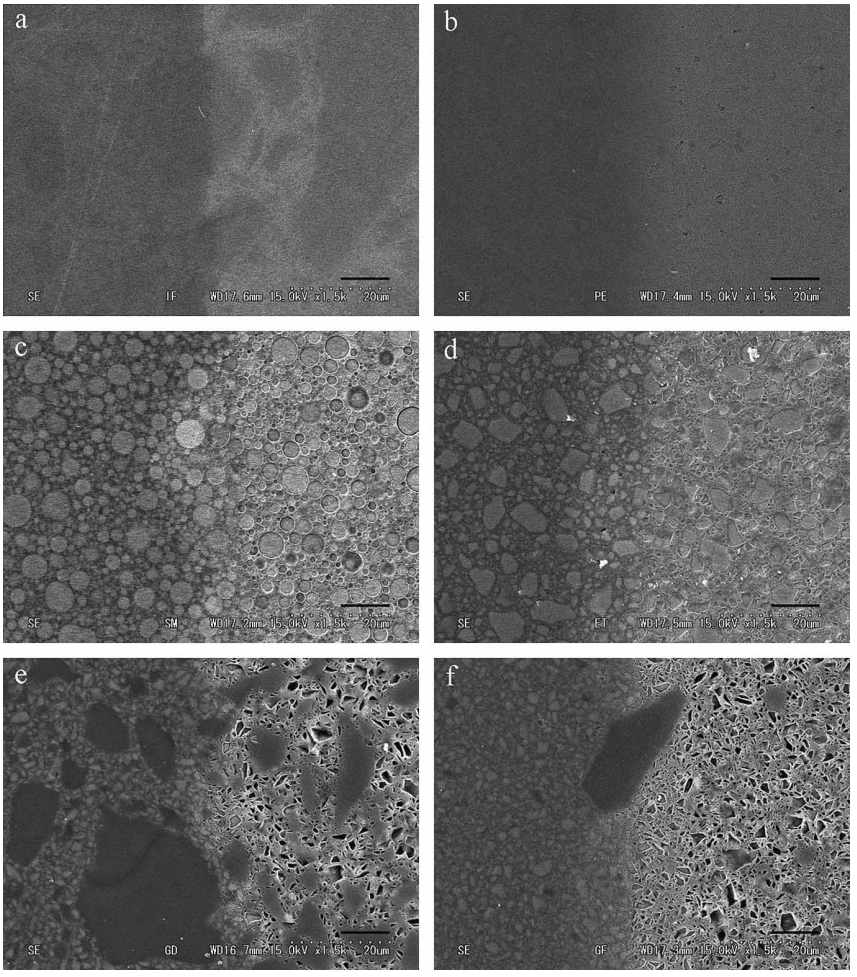

Figure 1. Microphotographs of representative surfaces of composite materials observed with a scanning electron microscope (1500 × magnification). On the right side is the APF-treated area and on the left side the untreated area protected with varnish. a, IF; b, PE; c, CE; d, ES; e, GR; f, GF; bar = 10 μm.

prepolymerized filler of these composites occupies greater areas (Figures 1e and 2a).

Filler dissolution by acidic food and drink has also been reported [30]. Consequently, the choice of material should correspond to the oral environment of the individual, because every material reacts differently under the influence of degradation factors. On the other hand, APF solution is used for the chemical surface treatment of ceramics prior to resin bonding [31,32] or repair of composite material [33]. The effect of etching is presumed to differ with the composition of the targeted material. Moreover, the surface properties of dental materials can affect adherence of early colonizing bacteria, biofilm formation, and esthetics. Evaluation of surface topography and quantitative determination of the surface

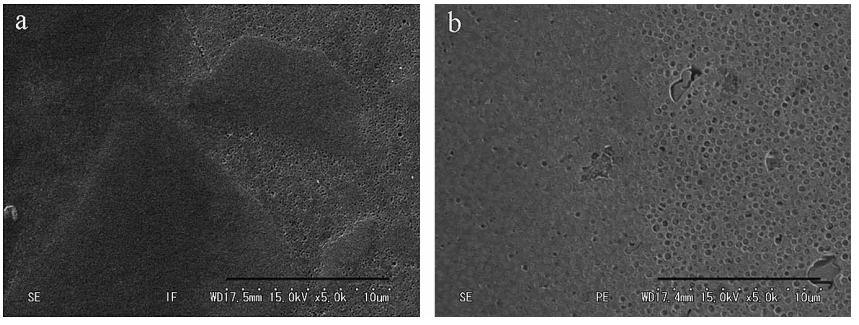

Figure 2. Microphotographs of representative surfaces of IF and PE observed with a scanning electron microscope at higher magnification (5000 × magnification). On the right side is the APF-treated area and on the left side the untreated area protected with varnish. a, IF; b, PE; bar = 10 μm.

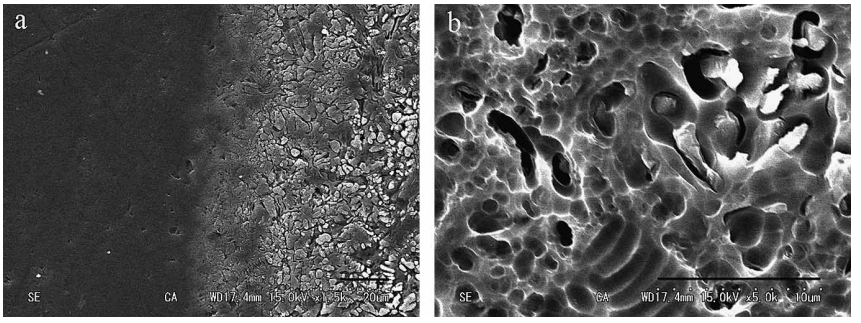

Figure 3. Microphotographs of representative surfaces of CAD observed with a scanning electron microscope. (a) On the right side is the APF-treated area and on the left side the untreated area protected with varnish (1500 × magnification); bar = 10 μm. (b) APF-treated area at higher magnification (5000 ×magnification); bar = 10 μm.

roughness may contribute to further studies. These results can be utilized to elucidate the relation of surface roughness to adherence of early colonizing bacteria or esthetic factors, and implant surface to cellular response in integrated tissue. The present study reveals a discrepancy among similar materials. Further studies are required to complete characterization of the surface properties.

In conclusion, confocal laser scanning and SEM analysis of prosthodontic composite materials subjected to APF application demonstrated evidence of acid attack on the exposed surface, and the responses varied with the material. Changes in surface roughness of microfilled material treated with APF were minimal. Gloss was strongly correlated to the surface roughness parameters, especially to the initial change in surface roughness. A stylus and a laser scanning device are both acceptable for measurement of the Ra parameter. However, for surface roughness, parameters such as Rz and RSm, the non-contact method properly describes various surface topographic features.

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