Surface Roughness of Nanofill and Nanohybrid Resin Composites after Polishing and Brushing

PISOL SENAWONGSE, DDS, MSc, PhD*
PONG PONGPRUEKSA, DDS, MSc*

ABSTRACT

Purpose: The purpose of this study was to compare the surface roughness of nanofill (Filtek Supreme XT dentin shade and transparent shade, Filtek Z350, and Estelite Sigma), nanohybrid (Tetric EvoCeram, Ceram X, and Premise), and microhybrid resin composites (Filtek Z250, Tetric Ceram, and Clearfil AP-X) with the materials after polishing or brushing.

Materials and Methods: Forty specimens of each resin composite were polymerized for 40 seconds under a matrix strip in cylindrical molds. Each type of polymer was divided into four subgroups: unpolished, polished with abrasive disks (Sof-Lex), polished with silicone-impregnated polishing devices (Astropol), and brushed with a toothbrush (Oral B) and toothpaste (Colgate). The surface roughness of each specimen was determined using a contact stylus profilometer and by observation under a scanning electron microscope.

Results: No significant differences in surface roughness among the materials were found on unpolished surfaces. For the nanofill resin composites, there were no significant differences in surface roughness between the two polishing methods or among the unpolished surfaces. After brushing, the surfaces of all materials, except those made from Filtek Z350 and Filtek Supreme XT (dentin), had greater roughness than unpolished surfaces and surfaces polished with either abrasive disks or silicone devices. The scanning electron microscope studies revealed that the surface irregularities of the materials corresponded to the results obtained using the surface roughness tester.

CLINICAL SIGNIFICANCE

The use of nanofill resin composites made with nanoclusters demonstrated the smoothest surfaces after polishing and brushing.

(*J Esthet Restor Dent* 19:265–275, 2007)

INTRODUCTION

Resin composites are widely used for the direct restoration of both anterior and posterior teeth because of the esthetic, physical, and mechanical properties of these materials. The surface properties of materials used in restorations are critical for their success because they mediate the interaction of restorative materials with the oral environment, such as bacterial accumulation.[1–3] Surface roughness is an important surface property. The surface roughness of a resin composite relates to the composition and porosity of the material and the instruments and procedures used in polishing.[4–8] In addition, the surface roughness of a resin composite has been recognized as a parameter of high clinical relevance for wear resistance, plaque accumulation, gingival

*Lecturer, Department of Operative Dentistry, Faculty of Dentistry, Mahidol University, Bangkok, Thailand

DOI 10.1111/j.1708-8240.2007.00116.x

inflammation, material discoloration (especially in Class V restorations), and surface gloss.[1–3]

Resin composite is a heterogenous material that is composed of three major components: resin matrix, filler particle, and silane coupling agent.[9] The resin matrix and filler particles have different levels of hardness that cause variations in removal efficiency after polishing; this variability can lead to differences in surface roughness. Because of composition diversity, various resin composites exhibit different levels of surface roughness after polishing. Materials with fillers of larger sizes generally show more surface roughness than those with fillers of smaller sizes.[5,6]

Resin composites have been classified according to various characteristics such as filler type, filler distribution, average particle size of filler, and physical and mechanical properties of the materials. Currently, three categories have been proposed for widely used resin composites: microfilled, microhybrid, and nanocomposite (nanofill or nanohybrid resin composite).[10] Nanofill is a composite resin that is composed of both nanomer and nanocluster, whereas nanohybrid is a hybrid resin composite with nanofiller in a prepolymerized filler (PPF) form. Microhybrid composites and nanocomposites are now well accepted for use on both anterior and posterior teeth as universal resin composites.

After restoration, removal of excess materials, recontouring, and surface polishing are generally performed. This finishing work has been reported to affect the roughness of the polished surfaces.[4,7] Currently, many finishing and polishing devices have been proposed such as coated abrasives (abrasive disks), cutting devices (carbide burs and stones), fine diamond burs, rubberized abrasives (resin impregnated or silicone-impregnated burs), and loose particulate abrasives (polishing paste).[11] The coated and rubberized abrasive devices are often clinically selected for finishing and polishing resin composites to a smooth and glossy appearance. Using different polishing devices has been demonstrated to result in variations of surface roughness after polishing.[4,7] Additionally, polished surfaces are often abraded by toothbrushing.[12]

The present study evaluated the surface roughness of microhybrid, nanofill, and nanohybrid resin composites after polishing or brushing, using a profilometer test and by observation under a scanning electron microscope. We tested first whether surface roughness differed among selected resin composites and, second, whether surface roughness differed depending upon if the resin composites were polished with one of the two polishing devices or were brushed.

MATERIALS AND METHODS

Experimental Design

This study was performed under a fully randomized 4 × 10 array experimental design. Ten resin composites were surface treated using one of the four protocols. The resin composites and surface treatment protocols are summarized in Table 1. The compositions of the selected materials are shown in Table 2.

Specimen Preparation

Each material was placed into a cylindrical silicone mold (5-mm diameter × 2-mm height) that was placed between two microscopic glass slides covered with transparent matrix strips. A constant pressure was applied to extrude the excess material. All specimens were polymerized for 40 seconds with a light-curing unit (Trilight, 3M ESPE, Seefeld, Germany) operated in standard mode and emitting more than 800 mW/cm^2, as measured with a radiometer (Model 100, Demetron Corp., Danbury, CT, USA). Forty specimens were prepared for each material. The specimens in each group were further divided into four treatment subgroups. In subgroup 1, 10 specimens were untreated and used as controls (surface against matrix or unpolished surfaces). In subgroup

TABLE 1. FACTORS EXAMINED IN THE PRESENT STUDY.

Factors	Type	Manufacturer	Batch Number
Resin composites			
1. Filtek Z250	Microhybrid	3M ESPE, St. Paul, MN, USA	20050406
2. Tetric Ceram	Microhybrid	Vivadent, Schaan, Liechtenstein	E37123
3. Clearfil AP-X	Microhybrid	Kuraray, Osaka, Japan	00816A
4. Filtek Supreme XT (transparent shade)	Nanofill	3M ESPE	20050722
5. Filtek Supreme XT (dentin shade)	Nanofill	3M ESPE	20050706
6. Filtek Z350	Nanofill	3M ESPE	EXM667
7. Estelite Sigma	Nanofill	Tokuyama, Tokyo, Japan	E401
8. Premise	Nanohybrid	SDS Kerr, Orange, CA, USA	417566
9. Tetric EvoCeram	Nanohybrid	Vivadent	H11363
10. Ceram X	Nanohybrid	Dentsply, Konstanz, Germany	0502001970
Surface treatment protocols			
1. Under matrix (Hawe transparent strip)	Mylar strip	Hawe Neos Dental, Bioggio, Switzerland	
2. Abrasive disk (Sof-Lex)	Aluminum oxide-coated disk —coarse (100 µm) —medium (40 µm) —fine (24 µm) —x-fine (8 µm)	3M ESPE	p050326
3. Silicone polishing systems (Astropol)	Caoutchouc, silicon carbide, aluminum oxide, titanium oxide —coarse (45 µm) —fine (1 µm) —x-fine (0.3 µm)	Vivadent	5567
4. Brushing (Oral B Contura and Colgate)	Toothbrush: regular, soft Toothpaste: sodium fluoride, tricosan, sorbitol, water, PVM/MA copolymer, sodium lauryl sulfate, hydrated silica, titanium dioxide, mica, etc.	Gillette Thailand, Bangkok, Thailand	183661

PVM/MA = polymethylvinylether-co-maleic anhydride.

2, 10 specimens were polished by hand using a series of four grades of abrasive disks. Each grade of abrasive disk was applied to the surface under dry conditions for 1 minute, using a slow-speed handpiece running at 12,000 rpm in one direction. In subgroup 3, 10 specimens were polished by hand with silicone-impregnated polishing devices. Three grades of silicone disks were used. Each grade of silicone disk was applied to the surface under dry conditions for 1 minute, using a slow-speed handpiece running at 12,000 rpm in one direction. In subgroup 4, the remaining 10 specimens were

TABLE 2. COMPOSITIONS OF SELECTED MATERIALS ACCORDING TO THE MANUFACTURER'S DATA.

Resin Composite	Type of Filler	Resin Matrix	% Filler by Weight (vol)	Mean Particle Size of Filler
Clearfil AP-X	Barium glass	Bis-GMA, UDMA, TEGDMA	89 (70)	1–3 µm
Tetric Ceram	Barium glass, silica dioxide, ytterbium trifluoride, bariumalumino-fluorosilicate glass	Bis-GMA, TEGDMA	79 (60)	0.7 µm
Filtek Z250	Zirconia/silica	Bis-EMA, UDMA, Bis-GMA	82 (60)	0.6 µm
Filtek Z350	ZrO2/SiO2 nanocluster, SiO2 nanofiller	Bis-PMA, DUDMA, Bis-GMA, TEGDMA	82 (60)	Nanocluster: 0.6–1.4 µm Nanofiller: 20 nm
Filtek Supreme XT (dentin shade)	ZrO2/SiO2 nanocluster, SiO2 nanofiller	Bis-PMA, DUDMA, Bis-GMA, TEGDMA	82 (60)	Nanocluster: 0.6–1.4 µm Nanofiller: 20 nm
Filtek Supreme XT (transparent shade)	ZrO2/SiO2 nanomer	Bis-PMA, DUDMA, Bis-GMA, TEGDMA	72 (57)	Nanofiller: 75 nm
Estelite Sigma	Silica/zirconia	Bis-GMA, TEGDMA	82 (71)	Spherical filler: 0.2 µm
Tetric EvoCeram	Barium glass, ytterbium trifluoride, PPF mixed oxide	Bis-GMA, TEGDMA	76 (61)	0.5 µm
Ceram X	Barium alumino-borosilicate glass, silica nanofiller, PPF	Methacrylate modified polysilane, dimethacrylate resin	76 (67)	Glass: 1 µm Silica: 0.02 µm
Premise	Barium alumino brosilicate glass, silica nanofiller, PPF, barium glass, discrete nanofiller	Bis-GMA, ethoxylated bisphenol-A-dimethacrylate, TEGDMA	84 (69)	Glass: 0.4 µm Silica: 0.02 µm

Bis-GMA = bisphenol-A-glycidyl methacrylate; Bis-EMA = bisphenol-A-ethoxylate glycidyl methacrylate; Bis-PMA = bisphenol-A-polyethylene glycol diether dimethacrylate; DUDMA = diurethane dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate; PPF = prepolymerized filler.

subjected to brushing with an Oral B toothbrush and Colgate Total toothpaste, using a brushing machine set at a load of 500 gf and a frequency of 80 strokes/min for 20,000 strokes. A diagram of the brushing device is shown in Figure 1. The specimen was placed into a silicone holder that itself was placed into the metal frame of the brushing machine. The specimen was brushed in a linear motion in a

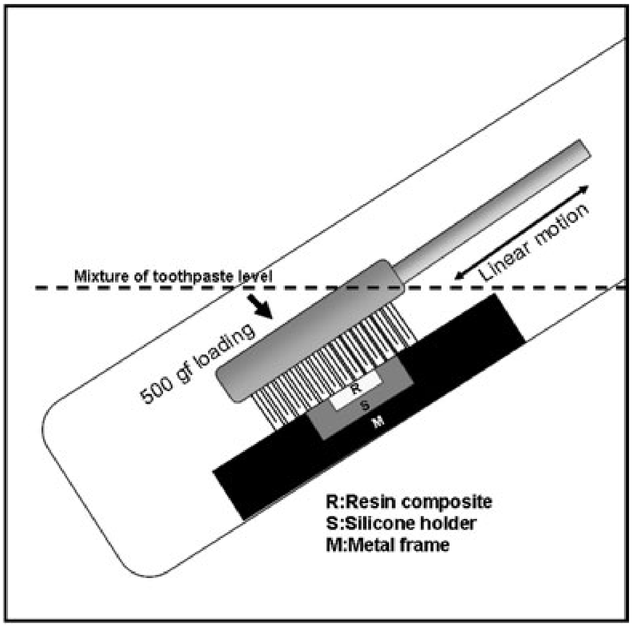

Figure 1. Diagram of brushing apparatus.

chamber containing a mixture of 50 g of toothpaste and 80 mL of distilled water.

Surface Roughness Test

The prepared specimens were subjected to a surface roughness test with a contact stylus profilometer (Talysurf series 2, Taylor Hobson Limited, Leicester, England) with a 2-μm diamond stylus employing a cutoff length of 0.25 mm, a measuring length of 2 mm, and a speed of 0.5 mm/s. Preliminary testing was performed to evaluate the specimens for defects (i.e., cracks, air bubbles) under a stereomicroscope at a magnification of 100×. The surfaces that were free from defects were tested by taking a reading at the center of each specimen. Three recordings were made per specimen surface. The measurements were taken perpendicular to the direction of polishing/brushing. The roughness parameter was evaluated as the arithmetic mean of the sum of the roughness profile values (*Ra*). The roughness means were recorded as the representative data value for each specimen. Statistical analysis was performed using two-way analysis of variance (ANOVA) and Dunnett's multiple comparisons at a 95% confidence interval.

Scanning Electron Microscope Observations

After performing the surface roughness test, the specimens were observed for surface irregularity under a scanning electron microscope (JSM 5410LV, Jeol, Tokyo, Japan) at a magnification of 750×.

RESULTS

Surface Roughness Study

The two-way ANOVA results are shown in Table 3. The analysis showed that there was a significant difference between the unpolished resin composites compared with the polished/brushed resin composites. The multiple comparison and mean roughness parameters obtained from the stylus profilometer are presented in Table 4.

Comparison of Surface Roughness among Materials

No significant differences in surface roughness among the unpolished materials were found. After polishing with abrasive disks, Clearfil AP-X demonstrated the greatest surface roughness. Filtek Z250 showed less roughness than Clearfil AP-X, but more roughness than the materials other than Tetric Ceram and Ceram X. No statistical differences in surface roughness among nanocomposites were found except between Filtek Z350 and Ceram X. After polishing with silicone-impregnated burs, Clearfil AP-X surfaces exhibited more roughness than all of the other groups, except for Tetric Evo-Ceram and Ceram X. No statistical differences were found among Tetric Ceram, Filtek Z250, Filtek Z350, Filtek Supreme XT, Estelite Sigma, and Premise. After brushing,

TABLE 3. TWO-WAY ANALYSIS OF VARIANCE OF SURFACE ROUGHNESS.

Source	Type III Sum of Squares	Degrees of freedom	Mean Square	*F*-value	Significance
Corrected model	4.736(a)	39	0.121	67.379	0.000
Intercept	3.560	1	3.560	1974.888	0.000
Material	0.715	9	0.079	44.076	0.000
Condition	2.857	3	0.952	528.389	0.000
Material × condition	1.164	27	0.043	23.923	0.000
Error	0.649	360	0.002		
Total	8.945	400			
Corrected total	5.385	399			

$R^2 = 0.880$. Selected materials (Filtek Supreme XT dentin shade and transparent shade, Filtek Z350, Estelite Sigma, Tetric EvoCeram, Ceram X, Premise, Z250, Tetric Ceram, and Clearfil AP-X). Polishing devices (Sof-Lex and Astropol)/brushing (Oral B Contura and Colgate).

the surfaces of Tetric EvoCeram and Ceram X were rougher than the other groups. The brushed surfaces of Filtek Z350 and Filtek Supreme XT (dentin shade [D]) were less rough than other groups.

Comparison of Surface Roughness among Polishing and Brushing Treatments

Post-treatment surface roughness for the nanofill resin composites did not differ significantly among the two polishing methods and the unpolished surface. For nanohybrid resin composites, only the Premise group showed no significant differences in surface roughness among the surfaces polished using the two polishing devices and the unpolished surfaces. Tetric EvoCeram specimens polished with silicone-impregnated burs demonstrated greater roughness than the unpolished surface, while Ceram X specimens polished with abrasive disks and silicone-impregnated burs also demonstrated more roughness than the unpolished surface. For microhybrid resin composites, Clearfil AP-X polished with abrasive disks and silicone-impregnated burs demonstrated more roughness than the unpolished surface. Z250 polished with abrasive disks also

TABLE 4. MEAN SURFACE ROUGHNESS (µM) OF RESIN COMPOSITES.

Resin Composite	Under Matrix	Abrasive Disk	Silicone Bur	Brushing
Microhybrid resin composites				
Clearfil AP-X	0.022 (0.005)[a,b]	0.145 (0.025)[f]	0.103 (0.049)[e]	0.299 (0.116)[i]
Tetric Ceram	0.024 (0.003)[a,b,c]	0.065 (0.015)[b,c,d,e]	0.054 (0.013)[a,b,c,d]	0.235 (0.074)[h]
Filtek Z250	0.017 (0.002)[a]	0.105(0.027)[e]	0.040(0.012)[a,b,c]	0.148(0.010)[f]
Nanofill resin composites				
Filtek Z350	0.017 (0.003)[a]	0.020 (0.003)[a,b]	0.020 (0.003)[a,b]	0.051 (0.009)[a,b,c,d]
Filtek Supreme XT (dentin shade)	0.016 (0.003)[a]	0.038 (0.017)[a,b,c]	0.038 (0.009)[a,b,c]	0.052 (0.016)[a,b,c,d]
Filtek Supreme XT (transparent shade)	0.020 (0.004)[a,b]	0.029 (0.005)[a,b,c]	0.038 (0.006)[a,b,c]	0.183 (0.081)[f,g]
Estelite Sigma	0.021 (0.002)[a,b]	0.054 (0.009)[a,b,c,d]	0.049 (0.041)[a,b,c,d]	0.369 (0.115)[j]
Nanohybrid resin composites				
Tetric EvoCeram	0.029 (0.009)[a,b,c]	0.051 (0.013)[a,b,c,d]	0.085 (0.017)[d,e]	0.428 (0.071)[k]
Ceram X	0.018 (0.003)[a]	0.069 (0.012)[c,d,e]	0.088 (0.010)[d,e]	0.411 (0.131)[k]
Premise	0.026 (0.003)[a,b,c]	0.057 (0.014)[a,b,c,d]	0.035 (0.009)[a,b,c]	0.207 (0.057)[g,h]

The numbers in parentheses are standard deviations. The data with the same superscript letters demonstrated no statistically significant differences.

demonstrated more roughness than the unpolished surface. Polishing Clearfil AP-X and Filtek Z250 with abrasive disks resulted in more surface roughness than when these materials were polished with the silicone-impregnated devices.

After brushing, the surfaces of all of the materials, except Filtek Z350 and Filtek Supreme XT (D), showed more roughness than the unpolished surfaces and surfaces subjected to polishing with abrasive disks or silicone-impregnated burs. Additionally, Filtek Z350 and Filtek Supreme XT (D) demonstrated fewer surface irregularities after brushing than the other materials. The highest values of roughness were observed after brushing of Tetric EvoCeram and Ceram X; the roughness values of these two materials after brushing did not differ from each other.

Scanning Electron Microscope Study

The scanning electron microscope studies revealed that the surface irregularities of the materials corresponded to the results of the surface roughness study (Figure 2). The data indicated relatively uniform surface topography before and after polishing in nanofill resin composites. Scratch lines from using abrasive disks could be observed in this group. Brushing increased surface irregularities in two materials of the nanofill group, but not for Filtek Z350 and Filtek Supreme XT (D).

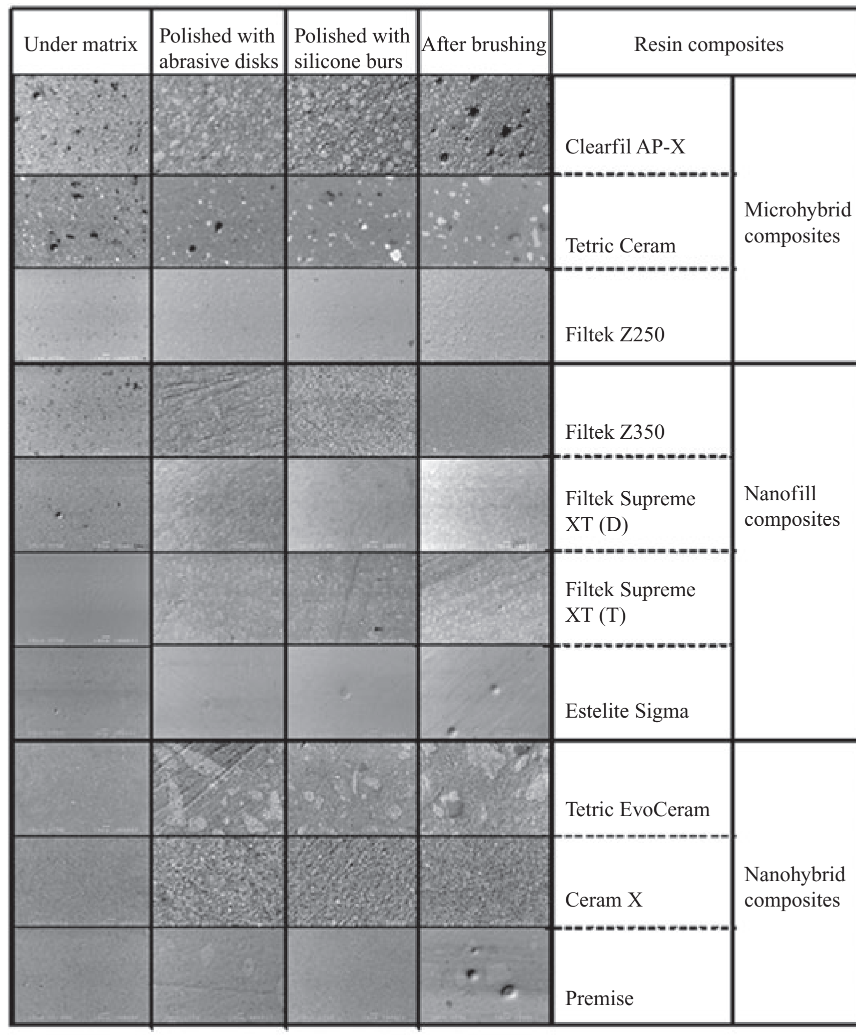

Figure 2. Scanning electron micrographs of the topographic surfaces of resin composites. D = dentin shade; T = transparent shade.

The scratch line from brushing could be observed in Filtek XT (transparent shade [T]), and surface dislodgement of silica-zirconia spherical fillers could be observed on Estelite Sigma after brushing. For microhybrid and nanohybrid resin composites, surface irregularities and filler dislodgement on the material surfaces were observed to varying degrees after polishing, except for Filtek Z250 and Premise. After brushing, all of the microhybrid and nanohybrid materials demonstrated surface irregularities and surface filler dislodgement.

DISCUSSION

Previously, the surface against matrix has been reported to be the smoothest surface for most direct tooth-colored restorations.[5] However, this surface is polymer-rich, making it relatively unstable.[13–15] The polymer-rich layer is often clinically removed by finishing and polishing,[16] which causes roughness on the polished surface to varying degrees depending on the polishing system and material used.[17–19] The smoothest polished materials should have a surface roughness comparable to the surface against matrix.[5]

In the present study, the microhybrid and nanohybrid resin composites were the smoothest surfaces against matrix.[5,17,18] These surfaces against matrix were smoother than polished surfaces because the unpolished surfaces are composed of more polymer matrix than fillers.[20] After polishing, these composites presented greater surface roughness than the unpolished surfaces to varying degrees. On the contrary, nanofill resin composites showed no significant differences in surface roughness among the two polished and unpolished surfaces. There may not have been a difference because a nanofill composite has an average particle size less than that of microhybrid or microfilled resin composites.[21]

Abrasive and silicone-impregnated disks affected the surface roughness of all microhybrid and nanohybrid resin composites, except for Premise. The various sizes of fillers in both the microhybrid and the nanohybrid resin composites exposed to the surface after polishing that were detectable in the scanning electron microscope studies could explain this result.[21,22] Clearfil AP-X, which was composed of the largest filler particle size in this study, had the highest roughness values after polishing with both systems. Similarly, for the nanohybrid resin composites, the higher levels of surface roughness correlate to the larger filler particle sizes as well. For example, Premise has a smaller filler particle size than other materials in the nanohybrid composite group, and this material had less surface roughness than the other materials in this group. The correlation between filler size and surface roughness has also been observed by others.[6]

It is not known whether the variability of surface roughness after polishing with the two systems used here is clinically relevant. It has been reported that the critical threshold value of *Ra*, to be clinically relevant, is 0.2 μm.[23] Over this value, surface roughness will increase plaque accumulation, risk of caries, and periodontal inflammation. As the roughness of all materials after polishing in the present study was less than 0.2 μm, clinical relevance of the roughness may not be unlikely.

In clinical situations, resin composites have to be able to withstand toothbrushing that causes wear to the materials.[24] Materials that can resist the wearing process and maintain a comparatively smooth surface when compared with the surface against matrix are preferred.[1–3] The present profilometer and electron microscope results indicate that, of the materials tested, only Filtek Supreme XT (D) and Filtek Z350 could be expected to withstand the wear caused by brushing. Only four materials—that is, Filtek Z250, Filtek Z350, Filtek Supreme XT (D), and Filtek Supreme XT (T)—exhibited *Ra* values less than 0.2 μm. Roughness values greater than 0.2 μm might result in a simultaneous increase in plaque accumulation and increased risks of secondary caries and periodontal inflammation.[23] The nanohybrid resin composites had higher roughness levels than did the nanofill resin composites. The scanning electron microscope images demonstrated the loss of PPF from the nanohybrid resin composites after brushing. The disruption of the filler matrix interface from the loss of PPF may explain the significantly greater *Ra* values observed. Dislodgement of nanoclusters from the nanofill resin composite, as has been reported in a previous study,[24] was not observed.

CONCLUSION

Treatment of different resin composites with polishing and brushing led to varying degrees of surface roughness, depending upon the polishing systems and materials used. Brushing caused increased roughness on all surfaces, and the effects of polishing and brushing were reduced when nanofill resin composites containing nanoclusters were applied. The results of the present study indicate that the nanofill resin composites with high filler loadings, such as Filtek Z350 and Filtek Supreme XT (D), best withstand the polishing and brushing processes. With comparable physical and mechanical properties as microhybrid resin composites, these nanofill resin composites have the potential to become good universal composite resin materials.

DISCLOSURE

The authors do not have any financial interest in the companies whose materials are included in this article.

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Reprint requests: Pisol Senawongse, Department of Operative Dentistry, Faculty of Dentistry, Mahidol University, 6 Yothi Street, Phayathai, Bangkok 10400, Thailand. Tel.: +66-2-644-8644 (ext. 3411); Fax: +66-2-354-8510; e-mail: dtpse@mahidol.ac.th