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Surface Properties of Dental Nanocomposites After Finishing With Rigid Rotary Instruments

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Clinical Relevance

Both nanofilled and nanohybrid dental restoratives appear to have relatively smooth surfaces when finished with tungsten carbide finishing instruments, and such instruments may be used as the final instruments when completing composite restorations.

SUMMARY

This study evaluated the surface characteristics of three nanoparticle resin composites (Z350, Heritage 7 Nanohybrid, and Ice) using profilometry and scanning electronic microscopy (SEM) after subjecting them to sequential finishing procedures using rigid rotary instruments. Fifteen 8 mm × 8 mm × 2 mm specimens were fabricated for each tested composite and subjected to one of the following finishing regimens: finishing with green stones followed by white stones, finishing with 45-μm diamond followed by 15-μm diamond, finishing with a 12-fluted carbide followed by a 20-fluted carbide. After finishing, the surfaces were subjected to profilometric testing. Eight parallel tracings were used to scan the finished surface of each specimen. For each recorded profile, the average roughness (R_a) and extreme value descriptor (R_{max}) were recorded. On completion of the profilometric testing, two specimens from each group were randomly selected for qualitative assessment by SEM. Profilometric data was analyzed using a one-way analysis of variance and post hoc Scheffe tests. The results demonstrated that the three control groups of the tested nanocomposites behaved as a coherent group with respect to surface

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roughness. With the exception of the nanohybrid Heritage 7 finished with the carbide instruments, all composites showed an increase in surface roughness.

The Z350 and Ice finished with the carbide finishing instruments showed no statistically significant increase in R_a, but both of these composites showed significantly rougher surfaces when finished with diamonds compared with controls. The nanohybrids Ice and Heritage 7 demonstrated significantly higher roughness in terms of R_{max} when finished with stones. The SEM correlated well with roughness measurements. The conclusion may be drawn that diamonds and stones produce unacceptable surfaces in terms of roughness on the tested nanocomposites whereas finishing carbides produce acceptable surfaces.

INTRODUCTION

The introduction of filler particles into dental composites, with some or all of the inorganic particles being in the nanoscale range, has led to an improvement in the physical properties of available restorative materials. Incorporating some proportion of smaller filler particles into a composite formulation generally increases the filler loading of the materials to a limiting point, with benefits to the properties of the material including a reduction in polymerization shrinkage, increases in the strength and modulus of elasticity, and improvement in the polishability.

More specifically, filler particles in the nanoscale range have dimensions below the wavelength of visible light, making them unable to absorb or scatter light. This offers a benefit of improvement in optical properties.[1] Additionally, the small particle sizes lend themselves to improved finishing and polishing procedures, which affect surface roughness, initial gloss after finishing and polishing procedures, and long-term retention of smoothness.[1-3] These properties also directly affect the optical properties of the finished restoration.

As with other classes of dental composite materials, for example, microfilled hybrids, finishing and polishing of nanocomposites are essential steps in the completion of a directly placed restoration. Adequate finishing and polishing are required to prevent surface discoloration and plaque accumulation and to optimize esthetics.[4-6] Polishing, with decreasing grits of silicon carbide or aluminum oxide coated flexible discs or silicone polishing points, is normally completed after finishing at high speeds with more aggressive armamentarium, such as finishing diamonds, multi-fluted finishing carbides, and green and white stones. Finishing and polishing procedures are complementary—both are done consecutively; however, because of variations in the extent and position of the restoration and the configuration of most commercially available polishing discs and points, some surfaces of the restoration may be left inadequately polished with the only surface treatment being finishing. Başeren[7] concluded that the use of polishing devices might not be useful clinically for areas that are not readily accessible.

It has been postulated that nanocomposites, with their smaller filler particles, allow for higher filler loading, which stabilizes such particles within the composite matrix, prevents undue loss of filler particles during finishing procedures, and results in surfaces that experience less surface detriment.[8] Thus, it is theoretically possible to complete these restorations with finishing alone. Although there is a plethora of evidence in the scientific literature regarding roughness and surface geometry of nanocomposites after finishing and polishing, there is a scarcity of evidence with respect to this class of composite with some or all of the filler particles in the nano-range being subjected to sequential finishing with rigid rotary instruments alone.

The aim of this study was to evaluate the surface characteristics of the selected nanoparticle resin composites after subjecting them to finishing procedures using profilometry and scanning electronic microscopy (SEM).

MATERIALS AND METHODS

A power analysis was performed to determine the optimum number of specimens required to calculate statistical significance. Three nanocomposites were used in this investigation: a true nanofilled restorative, Z350 (3M-ESPE, St Paul, MN, USA), and two nanohybrids, Heritage 7 Nanohybrid (IBT Med, Miami, FL, USA) and Ice (SDI, Bayswater, Victoria, Australia). Sixty specimens (8 mm × 8 mm × 2 mm) were made for each composite, using Teflon-coated stainless steel molds.

In each case, the mold was filled with the test material, the surface of the material was covered with a Mylar strip, and the specimen was cured using an Elipar Free Light 2 (3M-ESPE) with its tip in direct contact with the Mylar strip. Following the respective manufacturers' recommendations, Z350 and Ice specimens were cured for 20 seconds and the

Heritage 7 specimens for 40 seconds. Before and during preparation of the composite samples, the output of the curing light was measured with a radiometer (Demetron LED radiometer, Kerr Corporation, Orange, CA, USA) and gave a consistent reading of 890 mW/cm^2.

Fifteen specimens from each material were used as controls. The remaining 45 specimens were randomly divided into three groups of 15 each and subjected to one of the following finishing regimens using friction grip, rotary instruments mounted on a four-hole air-driven high-speed handpiece (Midwest Tradition, Dentsply, York, PA, USA):

1) Green stones followed by white stones (Dura Stones, Shofu Dental Corporation, San Marcos, CA, USA)
2) Diamond burs, fine (45 μm) followed by ultrafine (15 μm) (Swiss Tech, Coltene Whaledent, Cuyahoga Falls, OH, USA).
3) Tungsten carbide burs, 12-fluted followed by 20-fluted (Safe Ended Series, S.S. White, Lakewood, NJ, USA)

The finishing procedure was accomplished using a high-speed handpiece running at a maximum speed of 300,000 rpm using water coolant and hand pressure to mimic clinical conditions. One author (QM) was responsible for all finishing of specimens. The finishing instruments were changed after every five specimens. The application time for each instrument was 30 seconds. After finishing, the specimens were washed with a vigorous air/water spray and dried with clean compressed air using the air/water tip on the delivery cart of a dental chair (Adec Proforma, Adec Corporation, Newberg, OR, USA).

After finishing, the composite specimens were quantitatively assessed by profilometry using the Mitutoyo Surftest 401 surface roughness analyzer (Mitutoyo America Corporation, Aurora, IL, USA) with a cutoff of 0.25 mm, a transverse length of 1.6 mm, a sample length of 1.25 mm, and a vertical band width of 50 μm. Eight parallel tracings were used to scan the finished surface of each specimen with a side shift of 0.2 mm between tracings. For each recorded profile, the average roughness (R_a) and extreme value descriptor (R_{max}) were recorded.

Levene's test was used to demonstrate that the roughness values (R_a and R_{max}) were selected from a normal population with equal variances. Statistical analysis was carried out by a one-way analysis of variance and post hoc Scheffe tests (SPSS, version 16.0 IBM Corporation, Armonk, NY, USA).

Table 1: Roughness Values (R_a) of a Nanocomposite and Two Nanohybrids After Various Finishing Regimens (Common Superscript Letters Denote a Significant Difference, $p<0.05$, From the Control Values)

Finishing Regimen	Z350	Ice	Heritage 7 Nanohybrid
Control	0.1	0.08	0.4
Finishing stones	0.40	1.88 ^{b}	0.74
Diamonds	0.66 ^{a}	0.61 ^{b}	0.63
Multi-fluted carbides	0.41	0.46	0.34

On completion of the profilometric testing, two specimens from each group, were randomly selected for qualitative assessment by SEM (Philips SEM 515, Philips, Eindhoven, Netherlands). A 2 mm × 2 mm section was removed from a corner of each specimen and sputter-coated with gold using a Denton Desktop II sputtering system (Denton Vacuum LLC, Moorestown, NJ, USA) before SEM analysis. Photomicrographs were taken at 300× magnification. Depending on the number, size, and pattern of irregularities noted, the resulting photomicrographs were categorized into one of the following: 1) relatively smooth surface, 2) minor surface alteration, or 3) major surface alteration. Photomicrographs were also taken at 5000× magnification in an attempt to visualize any pattern of resin and filler alterations.

RESULTS

Profilometry

R_a *Measurements*—There was no significant difference between the roughness measurements for the unfinished surfaces of the nanofilled and nanohybrid materials ($p>0.05$).

All finishing regimens resulted in a change in the surface roughness compared with the control specimens (Table 1). A nanohybrid (Ice) and the nanofilled (Z350) both showed increases in the average surface roughness when treated with all finishing instruments, whereas the nanohybrid, Heritage 7, showed a decrease in surface roughness compared with the unfinished control when finished with carbide finishing burs.

Table 2: Roughness Values (R_{max}) of a Nanocomposite and Two Nanohybrids After Various Finishing Regimens (Common Superscript Letters Denote a Significant Difference, $p<0.05$, From the Control Values)

Finishing Regimen	**Z350**	**Composite Type *Ice***	**Heritage 7 Nanohybrid**
Control	2.10	2.13	3.20
Finishing stones	3.43	16.0 [b]	18.48 [c]
Diamonds	5.48 [a]	4.47	4.70
Multi-fluted carbides	3.90 [a]	3.73	4.24

Compared with the control specimens prepared under a Mylar strip, there was a significant difference between the R_a for Z350 finished with diamonds and Ice treated with finishing stones and diamonds ($p<0.05$). The surfaces of Ice finished with the stones were statistically rougher than those finished with the diamonds ($p<0.001$). None of the finishing regimens produced statistically significant variations in the roughness values of the Heritage 7 nanohybrid.

R_{max} Measurements—The R_{max} values for Z350 showed significant increases in roughness compared with the control specimens when finished with the diamonds ($p<0.001$) and carbides ($p=0.033$) (Table 2). Additionally, the diamond instruments produced statistically rougher surfaces compared with the stones ($p=0.014$).

Both Heritage 7 and Ice nanohybrids showed highly significant increases in roughness when finished with the stones ($p<0.0001$). The R_{max} values for the surfaces finished with the stones were significantly rougher than those finished with diamonds ($p<0.001$) and carbides ($p<0.001$).

SEM Analysis

Visual assessment with the use of the photomicrographs correlated well with the values obtained for R_a. SEM of the surfaces of the control samples and those of all the three materials finished with carbide burs at 300× magnification showed relatively smooth surfaces (Figures 1a through c and 2a through c).

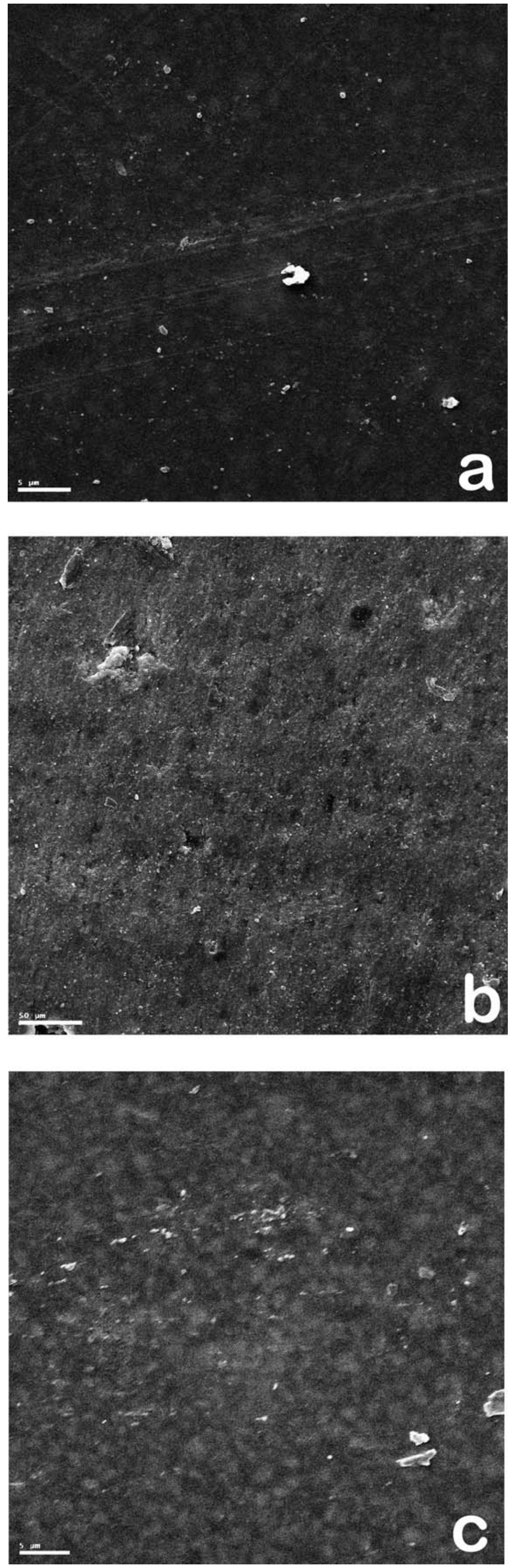

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Figure 1. *Smooth and homogenous surfaces of the (a): Z350, (b): Heritage 7, and (c): Ice control specimens. (300×).*

The nanofilled Z350 finished with the diamonds and observed at 300× magnification showed major surface alteration with distinct crests and grooves (Figure 3a). Interestingly, various degrees of surface alterations were noted; larger, more distinct troughs were seen parallel to the direction of finishing, and smaller striations appeared almost perpendicular to the main troughs, which was attributed to the regular spacing of diamonds on the body of the finishing bur. The pattern on the nanohybrids finished with the diamonds produced similar type of troughs, but these appeared to be shallower (Figure 3b,c).

Compared with the effect of the diamond instruments on the composite samples, sequential use of the green and white stones showed only minor surface alterations on both the nanofilled product and the two nanohybrids tested (Figure 4a through c). The widths of the troughs created were approximately one third the widths produced by the diamond instruments on analogous samples, and the distinct troughs were not as deep as those caused by the diamond instruments.

DISCUSSION

A typical profilometer consists of a stylus mounted on a lever arm capable of vertical movement. When the stylus is drawn a specified distance in contact with the test surface, its movement is translated into various parameters of surface roughness. The tracing generated by such movement consists of peaks and valleys, as shown in Figure 5. The parameter, R_a, is the average roughness, which is the mean of the peak-to-valley heights present in the test length. R_{max} is the maximum such value over the test length.

Several authors have used R_a as the main descriptor of surface characteristics of composite materials after finishing and polishing.[8-10] However, it has been suggested that the use of more than one roughness parameter is important when considering surface characteristics after more than one finishing instrument is used. [7,11]

As reported in many other studies of surface roughness of composite restoratives after finishing and polishing, the smoothest surfaces were those cured against Mylar strips. All three control groups of the evaluated composites behaved as a coherent

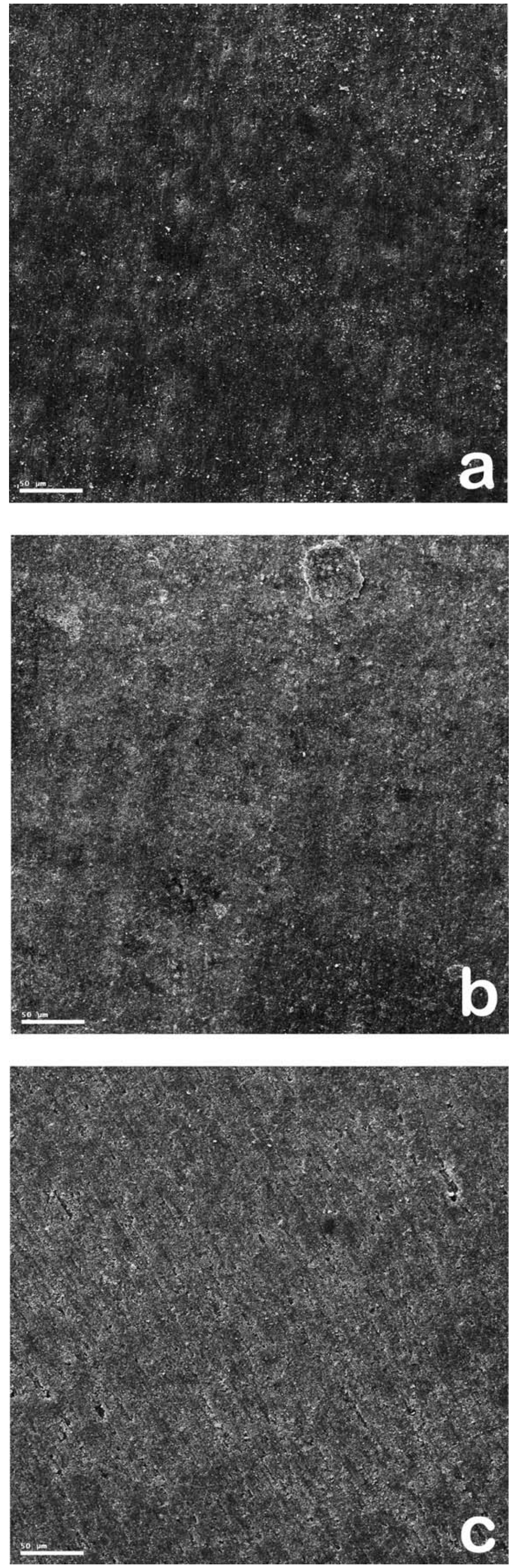

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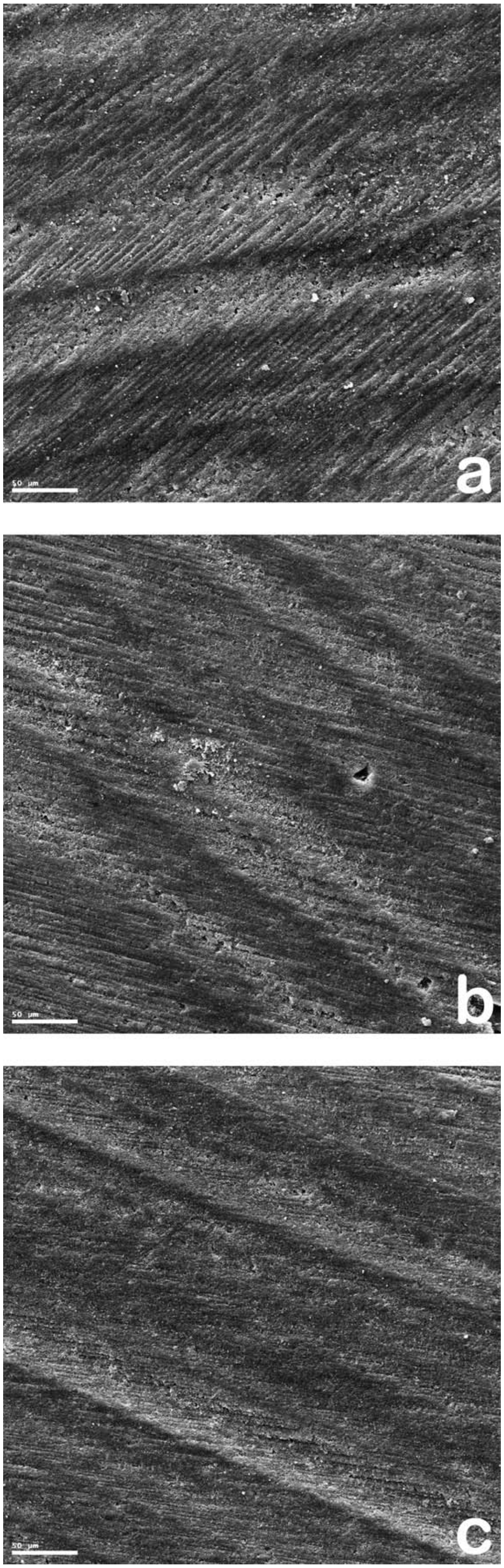
Figure 2. *Relatively smooth surfaces of the (a): Z350, (b): Heritage 7, and (c): Ice specimens after finishing with the 12-fluted and 20-fluted carbide burs. (300×).*



group when subjected to statistical tests; there were no statistical differences between them, even though the nanohybrid Heritage 7 demonstrated the largest absolute values for both R_a and R_{max}.

Across all the groups, multi-fluted tungsten carbide burs produced surfaces that were acceptable in terms of quantitative and qualitative assessment. The Heritage 7 specimens finished with the carbide instruments gave an unexpected result of a smoother surface compared with the control of the Heritage 7 group.

This is in contrast to the findings of Berastegui and others,[12] who concluded that both diamonds and finishing carbides should not be used as finishing instruments for composite restorations. However, other researchers have concluded that, because of the low cutting efficiency of finishing carbides, such instruments are best suited for finishing and smoothing hybrid composites.[13] In an SEM analysis of microfilled and hybrid composites, Ferracane and others[14] concluded that finishing carbides were less efficient at removing material. The results of the current study concur with statements regarding the nature of finishing carbides on composite materials regardless of the filler loading and predominant filler morphology. Indeed, SEM analysis of the nanocomposites finished with the multi-fluted finishing carbides tested demonstrated surfaces that looked almost planed or milled.

Diamonds appear to have a deleterious effect on the surface roughness of both the nanofilled Z350 and the nanohybrid Ice, as evidenced by the R_a values and SEM. Jung[13] has previously described the characteristic troughs that were observed in this study on other types of composites. He further concluded that finishing diamond instruments should be reserved only for gross removal and contouring because of their high cutting efficiency, a recommendation that could also be adopted for the finishing of nanocomposite materials.

Similarly, based on the R_{max} values and the SEM appearance obtained for the finishing stones, such instruments should not be used for the final polishing. From SEM analysis it appears that finishing stones are much more efficient at removing material from the surface of both nanofilled and

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Figure 3. *(a): Major surface alteration of Z350 nanofilled finished with a 45-μm finishing diamond followed by a 15-μm finishing diamond, with distinct troughs and striations noted perpendicular to troughs. Major surface alteration of (b): Heritage 7 and (c): Ice nanohybrid finished with a 45-μm finishing diamond followed by a 15-μm finishing diamond, with troughs more widely spaced. (300×).*

nanohybrid composites, and their cutting efficiency can be placed somewhat between that of the finishing carbides and diamonds.

The SEM micrographs of Z350 samples at higher magnification (5000×) explain the relatively high values of R_{max} with both the diamonds and carbide finishing burs. The larger particles of Z350 are actually loose agglomerations of nanoparticles, termed "nanoclusters." They are approximately 0.6-1.4 μm in size, behave as a single unit, and, according to the manufacturer, allow for filler loading of 59.5% by volume. Visualization of the troughs actually shows loss of some of these larger particles (Figure 6). This is in contrast to the manufacturer's claims that such nanoclusters are often not lost from the surface but, instead, in preference, the individual nanoparticles are lost from the cluster. Roeder and others[10] have discussed the effect of the reduced resin matrix phase on the dislodgement of larger filler particles during the finishing and polishing of the highly filled composites. They concluded that this is due to an inability to stabilize larger particles, with such large particles being plucked off during finishing and with increasing R_a values. Such an argument can be used for the nanocomposites tested in this study. The filler loadings, by volume, as supplied by the manufactures are 59.50% for Z350, 58.89% for the Heritage 7 nanohybrid, and 61.00% for Ice. Such highly filled composites, when subjected to the medium to high cutting efficiency of stones and diamonds, respectively, can lead to surface alterations that may not be compatible with esthetics or surrounding tissue health.

The differences in the SEM results between the nanofilled and the nanohybrid composites finished with the finishing diamonds could be explained by the relative hardness of the filler particle compared with the softer resin matrix. The Z350 has relatively hard zirconia/silica nanoparticles and nanosilica clusters compared with the softer bisphenol A glycidyl methacrylate (Bis-GMA), ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA), and urethane dimethacrylate (UDMA) resin matrix mixture, which facilitates preferential loss of resin compared with filler and gives the appearance of the deep troughs, almost regularly spaced defects. In contrast, the nanohybrid Ice contains strontium glass fillers with hardness values lower than that of zirconia/silicia nanoparticles.

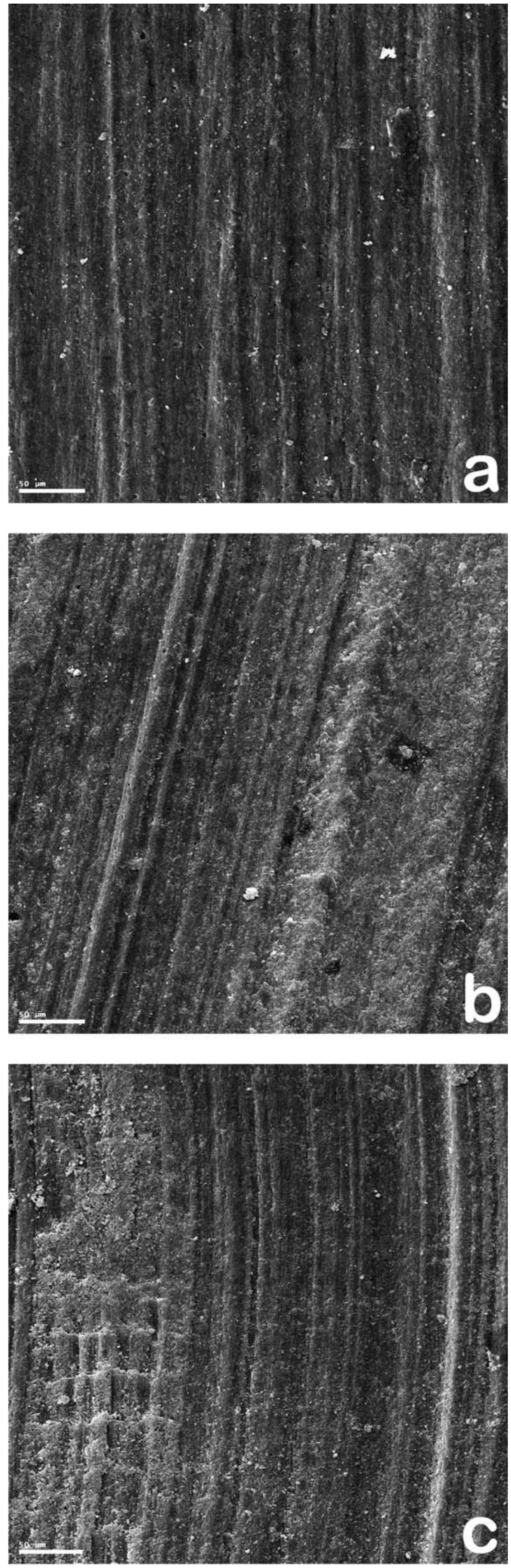

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Figure 4. *Minor surface alteration of (a): Z350, (b): Heritage 7, and (c): Ice specimens after finishing with green and white stones. (300×).*

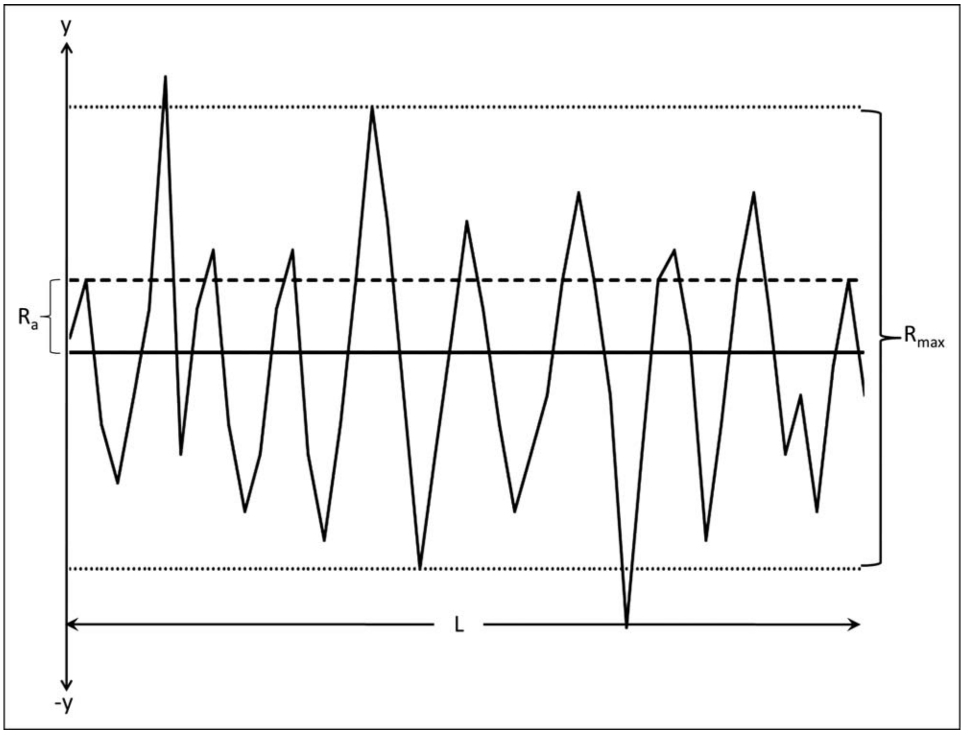

Figure 5. *A typical roughness plot.*

With the exception of the Ice finished with finishing stones, which produced an R_a value of 1.88, all the nanocomposites finished with the various regimens produced values within or below the range 0.7-1.0 μm. This is the range reported in the literature to have no appreciable increase in levels of plaque accumulation.[15,16]

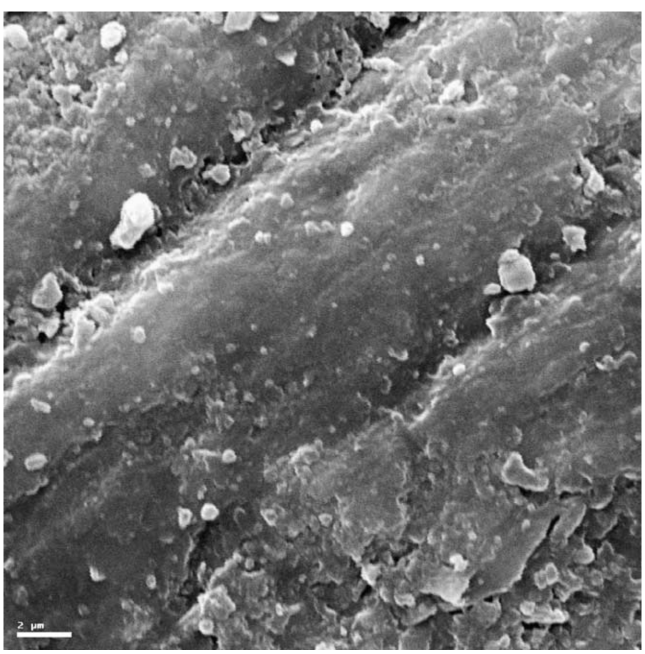

Figure 6. *High magnification SEM of the trough of Z350 nanofilled showing particle loss after finishing with a 45-μm finishing diamond followed by a 15-μm finishing diamond. (5000×).*

Although finishing with the multi-fluted carbide instruments produced acceptable results in terms of the surface properties, further research is warranted to compare these results with various classes of nanocomposites finished with carbide instruments and then polished with polishing disc, points, or pastes.

CONCLUSIONS

Within the limitations of this study the following conclusions may be drawn:

1) The nanofilled Z350 and the nanohybrids Ice and Heritage 7 had the smoothest surfaces when cured against Mylar strips.
2) The unfinished surfaces of all the tested composites behaved as a coherent group in terms of roughness measurements.
3) Carbide finishing instruments produced surfaces on all the tested composites that were comparable to their unfinished controls.
4) Finishing diamonds and stones are unsuitable for completing composites and are associated with significant surface detriment.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Mitra SB, Wu D, & Holmes BN (2003) An application of nanotechnology in advanced dental materials. *Journal of the American Dental Association* **134(10)** 1382-1390.
2. Yap AU, Low JS, & Ong LF (2000) Effect of food-simulating liquids on surface characteristics of composite and poly-acid modified composite restoratives. *Operative Dentistry* **25(3)** 170-176.
3. Silikas N, Kavvadia K, Eliades G, & Watts D (2005) Surface characterization of modern resin composites: a multitechnique approach. *American Journal of Dentistry* **18(2)** 95-100.
4. Lu H, Roeder LB, Lei L, & Powers JM (2005) Effect of surface roughness on stain resistance of dental resin composites. *Journal of Esthetic and Restorative Dentistry* **17(2)** 102-108.
5. Kawai K, & Urano M (2001) Adherence of plaque components to different restorative materials. *Operative Dentistry* **26(4)** 396-400.
6. Jefferies SR. (1998) The art and science of abrasive finishing and polishing in restorative dentistry. *Dental Clinics of North America* **42(4)** 613-627.
7. Başeren M (2004) Surface roughness of nanofill and nanohybrid composite resin and ormocer-based tooth colored restorative materials after several finishing and polishing procedures. *Journal of Biomaterials Applications* **19(2)** 121-134.
8. Jung M, Sehr K, & Klimek J (2007) Surface texture of four nanofilled and one hybrid composite. *Operative Dentistry* **32(1)** 45-52.
9. de Moraes RR, Gonçalves LS, Lancellotti AC, Consani S, Correr-Sobrinho L, & Sinhoreti MA (2009) Nanohybrid resin composites: nanofiller loaded materials or traditional microhybrid resins? *Operative Dentistry* **34(5)** 551-557.
10. Roeder LB, Tate WH, & Powers JM. (2000) Effect of finishing and polishing procedures on the surface roughness of packable composites. *Operative Dentistry* **25(6)** 534-543.
11. Jung M, Eichelberger K, & Klimek J. (2007) Surface geometry of four nanofiller and one hybrid composite after one-step and multiple-step polishing. *Operative Dentistry* **32(4)** 347-355.
12. Berastegui E, Canalda C, Brau E, & Miquel C (1992). Surface roughness of finished composite resins. *Journal of Prosthetic Dentistry* **68(5)** 742-749.
13. Jung M (1997). Surface roughness and cutting efficiency of composite finishing instruments. *Operative Dentistry* **23(3)** 98-104.
14. Ferracane JL, Condon JR, & Mitchem JC (1992). Evaluation of subsurface defects created during and finishing of composites. *Journal of Dental Research* **71(9)**1628-1632.
15. Shintani H, Satou N, Hayashihara A, & Inoue T (1985) Effect of various finishing methods on staining and accumulation of Streptococcus mutans HS-6 on composite resin. *Dental Materials* **1(6)** 225-227.
16. Weitman RT, & Eames WB (1975). Plaque accumulation on composite surfaces after various finishing procedures. *Journal of the American Dental Association* **91(1)** 101-106.