Effect of Nanofiller on Wear Resistance and Surface Roughness of Resin Composites

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Objective: *To compare the wear resistance and surface roughness of nanofiller-containing composites and microhybrid composites after simulated wear.*
Methods: *Five microhybrid composites and five nanofiller-containing resin composites were included in the study. Six cylindrical specimens with a diameter of 10 mm and a thickness of 6 mm for each material were prepared. The volume loss, vertical loss and the surface roughness (Ra) were determined after 800 cycles of simulated chewing motion. One specimen of each material was analysed by scanning electron microscopy (SEM) to compare the morphology of the wear surfaces. The microhybrid composites group and nanofiller-containing composites group were tested using the Mann-Whitney U test with a significance level of $\alpha = 0.05$.*
Results: *For all microhybrid composites, the average wear volume loss and vertical loss were 56.44 mm^3 and 730.6 μm, respectively, while the average wear losses of nanofiller-containing composites were 40.15 mm^3 and 528.17 μm, respectively. The nanofiller containing composite GNH400N showed the least roughness (Ra = 0.346 ± 0.076 μm), while the conventional microhybrid composite Ceramage showed the highest roughness (Ra = 0.699 ± 0.214 μm). However, wear resistance and surface roughness for the two groups showed no statistical difference. SEM micrographs of the nanofiller-containing composites after wear testing showed smoother and more uniform wear surfaces than for the microhybrid composites.*
Conclusion: *Nanofillers did not significantly influence the wear resistance of resin composites, but might improve the surface roughness of resin composites.*

Key words: *composite, microhybrid filler, nanofiller, roughness, wear*

Since Bowen[1] introduced dimethacrylate in the form of bis-GMA in 1962, the development of dental resin composites has gone through several stages, respectively characterised by macrofiller composites, microfiller composites, hybrid filler composites, microhybrid composites and nanofiller composites. Considerable improvements have been made in the properties of dental resin composites through these developments, which mainly focus on improvements in filler particle technology and chemical coupling[2,3]. The arrangement and geometry of fillers were optimised, whereby the wear resistance was improved. Some manufacturers claimed that the wear resistance of their resin composites could be comparable to that of amalgam.

In the past decade, nanotechnology has been widely applied in the dental area. Jandt et al[4] indicated that nanotechnology was the future development trend for resin-based dental materials. Recently, more and more dental nanofiller resin composites have been introduced onto the market. The manufacturers have claimed that those dental nanocomposites have superior material

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Table 1 Brand name, code, batch number and composition of tested materials

Material	Code	Batch	filler content (wt%)	Main composition	Manufacturer
Microhybrid composites					
Estenia C&B	ECB	0022BA	87.9	Bis-GMA, UDMA, UTMA, fine alumino-silicate glass particles (average particle size of 1.5 μm)	Kuraray Medical, Japan
Clearfil Majesty	CM	00221A	81	Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, pre-polymerised organic filler	Kuraray Medical
Ceramage	CMG	50994	73	UDMA, zirconium-silicate	SHOFU, Japan
Prime Art	PA	TL6	N/A	UDMA, TEGDMA, aromatic amine, silica, others	Sun Medical, Japan
Solidex	SD	70928	78	UDMA, organic filler, silica power	SHOFU
Nanofiller containing composites					
Twiny	TW	Trial Production	N/A	UDMA, TEGDMA, cramics cluster filler (1–6μm), sub-micro filler (200–600 nm), nanofiller (silica; 20–100 nm).	Yamakin, Japan
Luna-Wing	LW	1079922	68	Methacrylate monomer, inorganic filler	Yamakin
Gradia Forte	GF	909081	75	UDMA, silica power, prepolymerized filler	GC, Japan
GNH400N	GNH	Trial Production	80	N/A	GC
Filtek Supreme XT	FS	2E+07	78.5	Bis-GMA, bis-EMA, UDMA, TEGDMA, zirconia and silica particles (clusters of 0.6–1.4 μm, individual particle size of 5–20 nm)	3M ESPE, USA

UDMA, urethane dimethacrylate; UTMA, urethane tetramethacrylate; bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; bis-GMA, 2,2- bis[4(2-hydroxy-3-methacryloyloxy-propyloxy)-phenyl] propane; TEGDMA, triethylene glycol dimethacrylate; N/A, not available.

properties, such as excellent optical properties[5], easy handling characteristics, superior polishability[6], higher flexural strength, lower abrasion[7,8] and low polymerisation shrinkage[9,10]. However, after review studies on the properties of nanocomposites, there are still some controversies, especially regarding the wear resistance of nanocomposites[11-14]. A few studies showed that the nanofiller increased wear resistance of composites[11,13], but some showed this not to be the case[12,14]. The question of whether nanocomposite is superior in overall performance to conventional composites still needs further study.

The wear resistance and surface roughness of resin composites are very important factors to be considered when selecting these materials. However, clinical wear is a complex process, most likely involving several mechanisms, and is very difficult to simulate *in vitro*. Current wear machines can only simulate one or a few of the wear mechanisms, and no single device has been able to fully characterise resin composite performance in the mouth. Therefore, Heintze[15] suggested that it was reasonable to combine at least two different wear machines to assess the wear resistance of a new material. Although the wear resistance of nanofiller-containing composites has been evaluated by several wear-testing machines, e.g. OHSU[11], ACTA[12] and that using the tooth-brushing method[16], the CW3-1 wear machine, with a rubber plate as antagonist and fluorite powder as abrasive, has not previously been used to test these materials. Therefore, the purpose of this study was to compare the wear resistance of the microhybrid composites and nanofiller-containing composites by using the CW3-1 wear machine.

Nanofiller resin composite, such as Filtek Supreme[17], is composed of both discrete nanometre (20 nm and 75 nm) and nanocluster (0.6–1.4 μm) particles, whereas nanohybrid composite is a hybrid resin composite with nanofiller in a prepolymerised filler form[18]. In

this study, both nanofiller composites and nanohybrid composites are called nanofiller-containing composites.

Material and methods

A total of five microhybrid composites, Estenia C&B (ECB), Clearfil Majesty (CM), Ceramage (CMG), Prime Art (PA) and Solidex (SD), and five nanofiller-containing resin composites, Twiny (TW), Luna-Wing (LW), Gradia Forte (GF), GNH400N (GNH) and Filtek Supreme XT (FS), were included in this study. The detailed information for these materials is shown in Table 1.

Specimen preparation

For each material six cylindrical specimens with a diameter of 10 mm and a thickness of 6 mm were prepared. A standard split stainless steel mould was placed on a glass plate and filled with the resin composite layer by layer. Each layer, the thickness of which was no more than 2 mm, was light cured for 180 s using a polymerisation unit (Elipar^{TM} 2500, output: 550 mW/cm^2, 3M ESPE, St Paul, MN, USA). For the final layer, the mould was slightly overfilled, covered with a polythene film, pressed with another glass plate from the top to flush out the excess composite and light cured for 180 s. The cured specimen was then pushed out of the mould and stored in distilled water at 37°C for 24 h prior to testing.

Three-body wear

The wear resistance of the materials was evaluated using the CW3-1 wear machine (Peking University, Beijing, China), which has been described in detail in a previous study[12] (see Fig 1). The main characteristics of the CW3-1 wear machine are that the antagonist used was a rubber plate with a Shore hardness of 67 and a thickness of 6.5 mm and the abrasive used was a slurry of fluorite (fluorspar) powder mixed with distilled water.

The testing procedure for each specimen was as follows. The specimen was fixed on the specimen clip, and the grinding pool was filled with a mixture of 100 g fluorite powder (Mohs hardness 4, particle size 110–120 grit) and 25 g distilled water. The specimen was first subjected to 100 cycles of preliminary wear under a 5-kg load in order to eliminate the oxygen inhibition layer. Then 800 cycles of formal wear were conducted with a total load of 17 kg. Before and after the 800 cycles of formal wear, the specimen was washed for 5 min in an ultrasonic cleaner (SK3200LHC, KUDOS, Shanghai, China), dried using an air blower until there was no visible water on the specimen surface and then weighed

Fig 1 The CW3-1 wear machine.

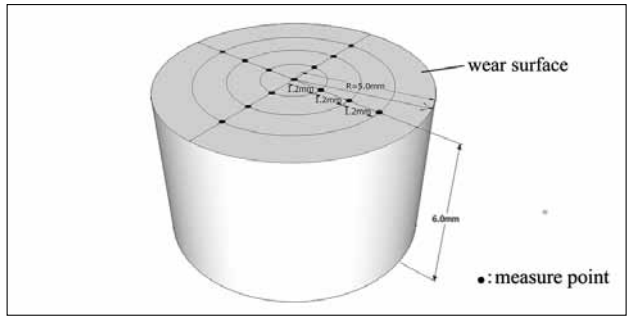

Fig 2 The measurement points on the specimen wear surface.

using a balance (XS105, Mettler Toledo, Columbus, OH, USA) with 0.01 mg accuracy. Weight loss was determined by comparing the weights of the specimen before and after the 800 wear cycles. The density was measured by means of a density meter (DT100, Beijing Optical Instrument Factory, Beijing, China). Finally, the volume loss was calculated. The vertical loss was determined by means of a dial indicator (ID-C112AM, Mitutoyo, Kawasaki, Japan; 0.001 mm) with a two-dimensional stepping apparatus before and after the 800 cycles of wear. Thirteen points on the wear surface were measured; the distribution of these points is shown in Fig 2. The average value of these 13 points represented the height of the wear surface.

Table 2 Volume loss, vertical loss and surface roughness of tested materials

Material	Volume loss (mean ± SD) (mm^3) $N = 6$	Vertical loss (mean ± SD) (μm) $N = 6$	Roughness (mean ± SD) (Ra) $N = 20$
ECB	28.68 ± 3.27	357.68 ± 28.88	0.432 ± 0.077
CM	56.46 ± 1.56	737.03 ± 22.71	0.542 ± 0.092
CMG	64.75 ± 1.91	852.26 ± 19.49	0.699 ± 0.214
PA	72.73 ± 2.05	935.41 ± 21.11	0.612 ± 0.102
SD	75.10 ± 3.76	963.24 ± 41.19	0.571 ± 0.116
TW	19.82 ± 1.03	258.63 ± 11.73	0.407 ± 0.043
LW	25.63 ± 1.46	336.25 ± 18.23	0.495 ± 0.071
GF	40.92 ± 1.38	544.41 ± 22.08	0.396 ± 0.095
GNH	45.09 ± 1.64	593.73 ± 17.43	0.346 ± 0.076
FS	53.74 ± 2.39	715.23 ± 28.29	0.523 ± 0.092

Measurement of surface roughness after wear testing

After simulated three-body wear, the roughness of two specimens of each material was measured using a contact stylus profilometer (SJ-400, Mitutoyo, Kawasaki, Japan) with a 2 μm diamond stylus. A measuring length of 2.5 mm and a speed of 0.5 mm/s were used. The roughness (Ra) of the defect-free wear surface was determined. Ten measurements were made per specimen surface. The roughness parameter for each specimen was evaluated as the arithmetic mean over the ten measurements.

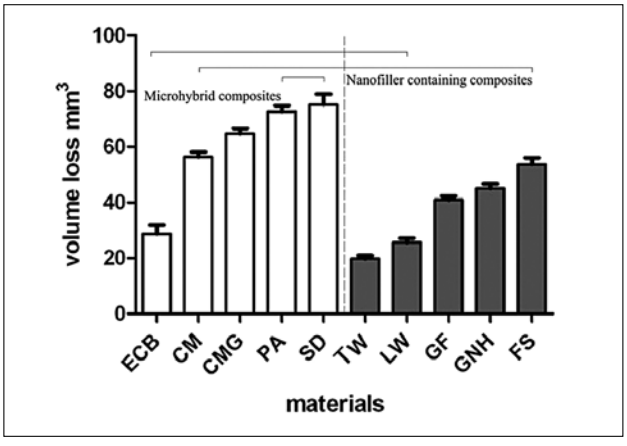

Fig 3 The volume loss of tested materials. The groups that are connected with a horizontal line are not significantly different from one another.

SEM evaluation

One specimen of each material was examined by SEM (DSM 950, Opton, Oberkochen, Germany) using secondary emission electron imaging at an accelerating voltage of 15 kV and ×2,000 magnification after gold sputtering using a JFC-1100 sputtering device (JEOL, Tokyo, Japan).

Statistical analysis

For each material, mean wear volume loss, vertical loss and surface roughness were analysed by one-way analysis of variance (ANOVA) ($\alpha = 0.05$). The microhybrid composites group and nanofiller-containing composites group were tested by the Mann-Whitney U test with a significance level of $\alpha = 0.05$. For all statistical evaluations, SPSS version 15.0 for Windows (Statistical Package for Social Science, SPSS Inc, Chicago, IL, USA) was used.

Results

Three-body wear

The material loss mean values and standard deviations after 800 wear cycles of all tested materials are presented in Table 2, Fig 3 and Fig 4.

The volume loss and vertical loss of microhybrid composites ranged from 28.68 mm^3 to 75.10 mm^3 and from 357.68 μm to 963.24 μm, respectively, and the

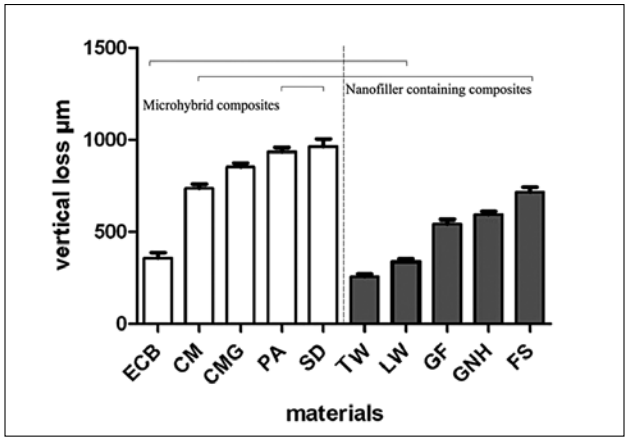

Fig 4 The vertical loss of tested materials. The groups that are connected with a horizontal line are not significantly different from one another.

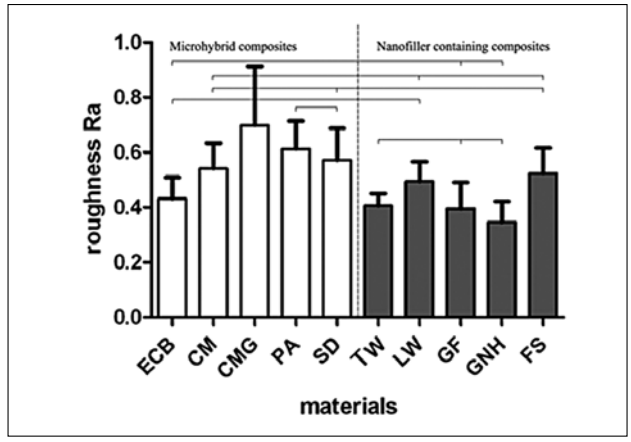

Fig 5 The surface roughness of tested materials after wear. The groups that are connected with a horizontal line are not significantly different from one another.

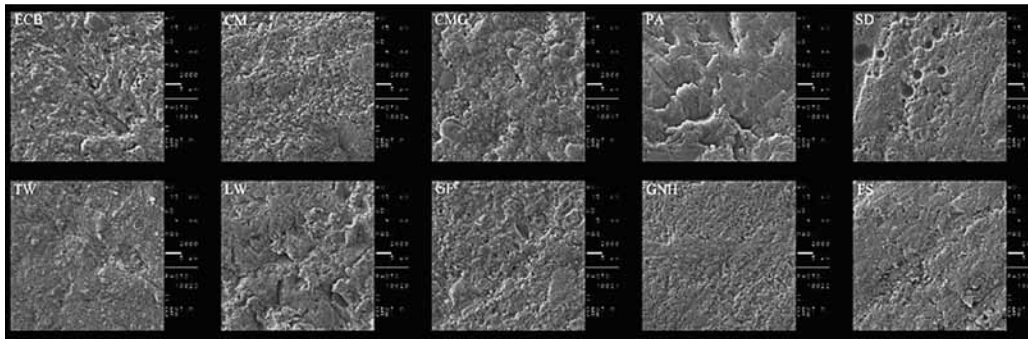

Fig 6 SEM micrographs of tested materials after 800 wear cycles (original magnification ×2,000). The upper five pictures are the microhybrid composites. The bottom five pictures are the nanofiller-containing composites.

corresponding values for nanofiller-containing composites ranged from 19.82 mm^3 to 53.74 mm^3 and from 258.63 μm to 715.23 μm, respectively. For the microhybrid composites, the average wear volume and vertical loss were 59.54 mm^3 and 769.12 μm, respectively, while those of nanofiller-containing composites were 37.04 mm^3 and 489.65 μm, respectively. One-way ANOVA showed significant differences in wear resistance between most of the materials. However, the Mann-Whitney U test showed that there was no significant difference in wear resistance between the two groups ($P > 0.05$).

Surface roughness

The mean values and standard deviations of surface roughness of the tested materials are summarised in Table 2 and Fig 5. One-way ANOVA showed that there were significant differences between some of the materials ($P < 0.001$). The nanofiller-containing composite GNH showed the lowest roughness (Ra = 0.346 ± 0.076 μm), and the conventional microhybrid resin composite CMG showed the highest roughness (Ra = 0.699 ± 0.214 μm). However, the Mann-Whitney U test showed no significant difference in roughness between the two groups ($P > 0.05$).

SEM evaluation

SEM micrographs of the tested materials after 800 wear cycles are presented in Fig 6. All of the nanofiller-containing composites showed a relatively smooth and uniform wear surface without significant filler protrusion or pits from filler dislodgement in the surrounding matrix, except LW. The microhybrid composites demonstrated more surface irregularities in the form of filler dislodgement and protrusion. The SEM micrographs of the nanofiller-containing composites after wear testing showed smoother and more uniform wear surfaces than the microhybrid composites.

Discussion

The wear resistance of resin composites is a very important factor to be considered when selecting composite materials for clinical use. Low wear resistance in a composite material induces loss of anatomical form, especially for large restorations, and is therefore considered a key factor limiting the clinical use of composites for posterior restorations[19,20]. However, there is still no generally accepted wear evaluation method[21]. The CW3-1 wear machine is one of the earlier successful laboratory wear-testing machines, developed by Xu et al[22] in the 1990s. The design and the parameters of the machine have previously been described in detail[22]. This machine can rapidly evaluate the wear resistance of a resin composite, which takes about 30 to 40 min for one specimen, with the mean coefficient of variation generally no larger than 5%. This is different from most other wear machines, which produce a mean coefficient of variation in material loss generally greater than 20%, with some reporting values as high as 70%[15,23]. Lower coefficients of variation indicate a lower relative variability, which in turn may indicate better reliability of the machine and higher discriminating power for different materials.

In 1978, Jorgensen and Asmussen[24] claimed that decreased inter-particle spacing improved the wear resistance of resin composite. This theory was further developed by Bayne et al[25] and confirmed by Schwartz et al[26] and Turssi et al[11]. In this present study, the average volume loss and vertical loss of nanofiller-containing composites were 37.04 mm^3 and 489.65 μm, respectively; lower than the corresponding values of 59.54 mm^3 and 769.12 μm for microhybrid composites. This is in accordance with Jorgensen and Asmussen's hypothesis. An explanation for the improvement in the wear resistance with the smaller particles is that the mean distance between neighbouring particles is smaller than with the larger filler particles. As a result, more particles will be present on the surface, providing better protection for the matrix. Another explanation for the improved wear resistance may be a stronger chemical integration of the nanoparticles with the resin matrix. Nanocomposites wear by the breaking of individual primary particles, rather than by the dislodgement of entire larger particles, and so the remaining worn filler particles can continue to provide protection to the matrix[5]. However, the difference in wear resistance between the two groups, nanofiller-containing and microhybrid composites, was shown by the Mann-Whitney U test not to be significant. This may be attributed to the large scatter of wear data within the groups, each of which contained several different materials. From Table 2, it can be seen that the volume loss and vertical loss of nanofiller-containing composites ranged from 19.82 mm^3 to 53.74 mm^3 and from 258.63 μm to 715.23 μm, respectively, while those of microhybrid composites ranged from 28.68 mm^3 to 75.10 mm^3 and from 357.68 μm to 963.24 μm, respectively. The highest wear loss was nearly three times the lowest wear loss from the same group.

Clinical studies are the gold standard for evaluating the properties of a new material. Palaniappan et al[27] conducted a 3-year randomised clinical trial to evaluate the clinical performance of nanocomposite (Filtek Supreme) versus a microhybrid composite (Z100, 3M ESPE, USA). The material loss through wear was measured by a 3D laser scanning device and the reported vertical loss of the nanocomposite (Filtek Supreme) and the microhybrid composite (Z100) were 75 ± 27 μm and 64 ± 26 μm, respectively, after 3 years of clinical service. However, there were no significant differences between the two materials for other evaluative indices considered, including wear. In a 2-year clinical evaluation of a nanofiller and a fine-particle hybrid resin composite using the Ryge criteria[28] and a 4-year clinical evaluation of a nanohybrid and a fine hybrid composite using the modified USPHS (United States Public Health Service) criteria[29], none of the evaluative indices showed any significant difference between the two groups. The results of these clinical studies are therefore in agreement with the results of the present study when analysed using the Mann-Whitney U test.

In addition to the filler particle size, the filler particle shape, the filler composition and the filler distribution are also important factors for the wear resistance of composites[2,11]. Therefore, it is difficult to predict the wear resistance of a material simply from its particle size.

The surface roughness is an important surface property of resin-based restorative composites. It has been recognised as a parameter of high clinical relevance for wear resistance, plaque accumulation, gingival inflammation, material discolouration and surface gloss[30]. *In vivo* and *in vitro* studies have shown significantly better polishability for nanofiller-containing composites than microhybrid composites[18,27,31]. The better polishability may be attributed to the use of nanofillers in such resin composites: the smaller the filler size, the lower the degree of filler pluck-out, and hence the better the polishability[31]. The SEM observations in our study revealed that the microhybrid composites demonstrated a higher level of surface filler dislodgement and protrusion, leading to a higher surface roughness. The nanofiller-containing composites, except LW, showed a

relatively smoother and more uniform wear surface than did the microhybrid composites. Protruding nanocluster particles were not found in the nanofiller-containing composites, which agreed with the results of Yap et al[32]. The rougher wear surfaces of LW may be associated with its larger filler and cluster sizes. It can be seen in Fig 6 that there are many traces of larger filler dislodgement on the wear surfaces of the LW samples. The size and morphology of the filler particles of LW should be studied further.

However, the quantitative roughness data measured by the profilometer did not correspond to the qualitative investigation using SEM. Moreover, the roughness data (Ra) of the test materials ranged from 0.346 to 0.699 μm, which was a greater range than reported elsewhere[18,31,33]. This may be attributed to the accelerated wear testing method used in the present study: the rapid wear of the specimens led to many buffing marks on the wear surfaces, which may have interfered with the profilometer's roughness measurement.

Conclusions

Nanofillers did not significantly influence the wear resistance of resin composites, but they may improve the surface roughness of resin composites.

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