

Comparison of occlusal wear of bulk-fill and conventional flowable resin
composites based on simulated wear tests and SEM observations

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Contents

| | |
|------------------------------|--------------|
| Summary | P. 1 |
| Introduction | P. 4 |
| Materials and methods | P. 5 |
| Results | P. 8 |
| Discussion | P. 9 |
| Conclusions | P. 12 |
| References | P. 13 |
| Tables and Figures | P. 15 |

This thesis is based on the published article listed below with additional data.

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Summary

The continuing development of flowable resin composites, including bulk-fill flowable resin composites, has led to expanded clinical applications of these resin composites in posterior lesions. Based on previous investigations, the clinical use of flowable resin composites in posterior teeth is currently increasing. Resin composite restorations in posterior teeth are subjected to a wide range of external forces, such as mastication and unconscious bruxism. These occlusal forces may also cause roughening of the restorative surfaces, leading to loss of anatomical form. Thus, the wear resistance of a resin composite is central to the long-term stability of posterior restorations. However, there is a few researches investigating the occlusal wear of bulk-fill and conventional flowable resin composites, despite the increasing usage of these materials for posterior restorations. Therefore, the investigation of the simulated occlusal wear of bulk-fill and conventional flowable resin composites is important in assessing whether such types of resin composites are genuinely able to sustain the wear that they encounter in clinical use. The purpose of this study was to compare the simulated occlusal wear between bulk-fill and conventional flowable resin composites.

Four bulk-fill flowable resin composites: Filtek Bulk Fill Flowable Restorative (FB), G-ænial Bulk Injectable (GB), SDR Flow+ (SD), and Tetric EvoFlow Bulk Fill (TB) were evaluated. Four flowable resin composites: Clearfil Majesty IC (CM), Filtek Supreme Ultra Flow (FF), G-ænial Universal Flow (GF), and Herculite XRV Ultra Flow (HF) were also evaluated. Twenty specimens of each of the bulk-fill and conventional flowable resin composites were prepared for simulated occlusal wear testing. Cylinder-shaped custom stainless steel fixtures were machined with a cylindrical cavity 6.5 mm in diameter and 4 mm in depth. A single 4 mm filling of the bulk-fill resin composites was cured for 40 s and two increments of the conventional flowable resin composites (approximately 2 mm in thickness) for 40 s each with a visible light 800 curing unit set at 600 mW/cm². After 24 h, the resin

composite surfaces were polished flat to #4,000-grit surface using a sequence of silicon carbide papers. Leinfelder-Suzuki (Alabama) wear simulation device was used in this investigation. The wear antagonists were stainless steel ball bearings mounted inside a collet assembly. During the application of the load, the antagonists rotate approximately 30° as the maximum force is reached (maximum load of 78.5 N at a rate of 2 Hz), and then counter-rotate back to the original starting position as the load relaxes to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulation device.

Prior to occlusal wear simulation, each resin composite specimen was profiled using a noncontact optical profilometer with a built-in software. Wear measurements were determined from differences between the before and after data sets. Maximum depth (MD; μm) and volume loss (VL; mm^3) of the wear facets were determined for the occlusal wear simulation for each of the eight resin composites.

To observe filler size, shape, and distribution in the bulk-fill and conventional flowable resin composites, cured specimens were mirror-polished. The polished surfaces were subjected to argon-ion beam etching for 40 s, and then SEM observations were performed. In addition, SEM observations of specimens after occlusal wear tests were also performed.

One-way ANOVA for both VL and MD showed a significant difference for the factor of material. Tukey's post-hoc test for VL and MD showed significant differences in simulated occlusal wear among the materials tested. VLs of the materials evaluated in this study ranged from 0.025 ± 0.005 to $0.148 \pm 0.033 \text{ mm}^3$. VLs of GF, GB, and FF were significantly less than the other five materials evaluated in this study. MDs for the materials in this study ranged from 98.1 ± 20.5 to $210.6 \pm 27.8 \mu\text{m}$. GF exhibited the least amount of wear among the eight materials evaluated and the rank order of simulated wear was GF–GB–FF–FB–CM–HF–TB–SD.

SEM observations of the cured resin composites after argon ion-etching showed that shape, size, and distribution of the filler particles were material dependent. FB and FF both employed nanosized spherical particles, and also exhibited aggregates of filler particles from 0.5 to 5 μm . GB and GF showed similar morphological features in that both resin composites employed densely packed nanosized irregular filler particles. SD exhibited irregular filler particles with a wide range in size, from 0.1 to 20 μm . TB and HF had relatively larger irregular filler particles and aggregated filler particles. CM exhibited irregular filler particles and somewhat larger pre-polymerized filler particles.

SEM images clearly showed that the morphological appearance of the wear facets were material and location dependent. Among the bulk fill resin composites, SD and TB showed rougher surfaces and larger facets as compared to those exhibited by the other bulk-fill resin composites. Although GB showed smaller and shallower wear facets than the other bulk-fill resin composites, the center of the facet showed some deep cleavages. Among the flowable resin composites, although the wear facets of CM, FF, and HF were smaller than those of bulk-fill resin composites with the exception of GB, the surfaces were somewhat rough. The wear pattern of GF was similar to GB, that is, the wear facet was small and shallow, but deep cleavages were observed at the center of the facet.

The results of this study showed that the simulated occlusal wear rates of GF, GB and FF were lower than those of the other tested resin composites, and the wear patterns were material dependent. The wear resistance of bulk-fill resin composites appeared to show an extremely broad range, much wider than that of the conventional flowable resin composites. Some resin composites of each type might be suitable for use in occlusal contact areas of posterior restorations, but care should be taken for selection of suitable materials.

Introduction

Flowable resin composites were first introduced in the late 1990s, and their handling properties and direct application systems eliminated some obstacles encountered when placing resin composite in small, narrow or complex cavities in inaccessible areas (1). Even though flowable resin composites became popular after launching into the dental market, the clinical applications of the first generation of flowable resin composites were limited to uses such as cavity liner, fissure sealant, or small cavities in posterior lesions due to their inferior mechanical properties compared to paste type of resin composites (2). Despite many improvements in these composites over twenty years, a survey of the clinical usage of flowable resin composites in posterior teeth found that most clinicians still using flowable resin composite only as cavity liner (3).

However, the continuing development of flowable resin composites, including bulk-fill flowable resin composites, has led to expanded clinical applications of these resin composites in posterior lesions due to improved mechanical properties based on advanced technologies, such as formulation modifications, increased filler loading, optimization of filler particle size, improved resin monomers, and modified filler surface treatment (4). Sumino *et al.* (5) reported that the mechanical properties of several flowable resin composites were comparable with paste-type resin composites. In addition, clinical studies over two years reported by Lawson *et al.* (6), and over three years reported by Kitasako *et al.* (7) found that flowable resin composites used in posterior teeth had similar clinical efficacy when compared to paste-type resin composites. Based on the results of these studies, the clinical application of flowable resin composites in posterior teeth is currently increasing.

Resin composite restorations in posterior teeth have been subjected to a wide range of external forces, such as mastication and unconscious bruxism (8). If the forces applied to resin composite restorations exceed the mechanical properties of the material itself, wear may occur,

and this might be particularly likely to happen in patients who apply greater than average forces during mastication (9). These occlusal forces may also cause roughening of the restorative surfaces, leading to loss of anatomical form (10). Thus, the wear resistance of a resin composite is central to the long-term stability of posterior restorations (11).

However, there is a few researches investigating the occlusal wear of bulk-fill and conventional flowable resin composites, despite the increasing usage of these materials for posterior restorations. Therefore, the investigation of the simulated occlusal wear of bulk-fill and conventional flowable materials is important in assessing whether such types of resin composites are genuinely able to sustain the wear that they encounter in clinical use.

The purpose of this study was to compare the simulated occlusal wear between bulk-fill and conventional flowable resin composites. The null hypothesis to be tested was that the simulated occlusal wear of bulk-fill and conventional flowable resin composites would not be influenced by the type of material.

Materials and methods

Study materials

Four bulk-fill flowable resin composites: 1) Filtek Bulk Fill Flowable Restorative (FB; 3M Oral Care, St. Paul, MN, USA); 2) G-ænial Bulk Injectable (GB; GC, Tokyo, Japan); 3) SDR Flow+ (SD, Dentsply Sirona, Milford, CT, USA); and 4) Tetric EvoFlow Bulk Fill (TB; Ivoclar Vivadent, Schaan, Lichtenstein), and four flowable resin composites: 1) Clearfil Majesty IC (CM; Kuraray Noritake Dental, Tokyo, Japan); 2) Filtek Supreme Ultra Flow (FF; 3M Oral Care); 3) G-ænial Universal Flow (GF; GC); and 4) Herculite XRV Ultra Flow (HF; Kerr, Orange, CA, USA) were evaluated in this study (Table 1).

Specimen preparation

Twenty specimens of each of the bulk-fill and conventional flowable resin composites were prepared for simulated occlusal wear testing. Cylinder-shaped custom stainless steel fixtures were machined with a cylindrical cavity 6.5 mm in diameter and 4 mm in depth. A single 4 mm filling of the bulk-fill resin composites was cured for 40 s and two increments of the conventional flowable resin composites (approximately 2 mm in thickness) for 40 s each with a visible light curing unit (Spectrum 800; Dentsply Sirona) set at 600 mW/cm². After 24 h, the resin composite surfaces were polished flat to #4,000-grit surface using a sequence of silicon carbide papers.

Wear simulation

Leinfelder-Suzuki (Alabama) wear simulation device was used in this study (Fig. 1). The wear simulator has a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive media (water slurry of unplasticized PMMA with average particle size of 44 μm). The media was placed inside the brass cylinders to cover the surface of the resin composite in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6 mm in height over the surface of the resin composite.

The wear antagonists were stainless steel ball bearings ($r = 2.387$ mm) mounted inside a collet assembly. The collet assemblies with the antagonists were then mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists rotated approximately 30° as the maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz), and then counter-rotate backed to the original starting position as the load relaxes to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulation device, over a period of about 55 h.

Wear measurements

Prior to occlusal wear simulation, each resin composite specimen was profiled using a noncontact optical profilometer (Proscan 2100; Scantron Industrial Products, Taunton, UK) with a built-in software. These profiles provided the pretest digitized contours (20 test specimens each for the eight resin composite materials for occlusal wear testing).

The X, Y, and Z coordinates of the before and after scans from the software were exported to another computer for analysis with AnSur 3D (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA) software. The X, Y, and Z coordinates generated with the software were saved as PRN files and then imported into the AnSur 3D program.

Wear measurements were determined from differences between the before and after data sets. A computerized fit was accomplished with the before and after data sets in the AnSur 3D, and maximum depth (MD; μm) and volume loss (VL; mm^3) of the wear facets were then determined for the occlusal wear simulation for each of the eight resin composites. A one-way analysis of variance (ANOVA) and Tukey's post hoc test were used for data analysis of VL and MD.

Scanning electron microscopy (SEM) observations

To observe the filler size, shape, and distribution of the bulk-fill and conventional flowable resin composites, the cured specimens were polished to a high gloss with abrasive discs (Fuji Star Type DDC; Sankyo Rikagaku, Saitama, Japan) followed by a series of diamond pastes down to a particle size of $0.25 \mu\text{m}$ (DP-Paste; Struers, Ballerup, Denmark). The mirror-polished surfaces were further subjected to argon-ion beam etching (IIS-200ER; Elionix, Tokyo, Japan) for 40 s, with the ion beam perpendicular to the polished surface (accelerating voltage = 1 kV; ion current density = 0.4 mA/cm^2). Subsequently, the surfaces were coated with a thin

gold film in an automatic ion sputter (Type SC-701; Sanyu Electron, Tokyo, Japan). Observations were performed using SEM (FE-8000; Elionix) at an operating voltage of 10 kV.

Observations for specimens after occlusal wear tests were also performed using SEM (TM3000; Hitachi-High Technology, Tokyo, Japan). For the specimens, a thin coating of gold-palladium alloy was applied in a sputter coater (Emitech SC7620 Mini Sputter Coater; Quorum Technologies, Ashford, UK). These observations were performed at an operating voltage of 15 kV.

Results

Simulated wear

The results of simulated occlusal wear (VL and MD) are presented in Table 2 and Fig. 2. One-way ANOVA for both VL and MD showed a significant difference ($p < 0.05$) for the factor of material. Tukey's post-hoc test for VL and MD showed significant differences in simulated occlusal wear among the materials tested. VLs of the materials evaluated in this study ranged from 0.025 ± 0.005 to 0.148 ± 0.033 mm³. VLs of GF, GB, and FF were significantly less than the other four materials evaluated in this study. MDs for the materials in this study ranged from 98.1 ± 20.5 to 210.6 ± 27.8 μm. GF exhibited the least amount of wear among the eight materials evaluated and rank order of simulated wear was GF-GB-FF-FB-CM-HF-TB-SD in this study.

SEM observations

Representative SEM images of the highly polished specimens of the eight resin composites after argon-ion etching are shown in Figs. 3A-H. FB and FF (Figs. 3A, F) both employed nanosized spherical particles, and also exhibited aggregated filler particles from 0.5 to 5 μm in size. GB and GF (Figs. 3B, G) showed similar morphological features in that both resin composites employed densely packed nanosized irregular filler particles (< 1 μm). SD

(Fig. 3C) exhibited irregular filler particles with a wide range in size, from 0.1 to 20 μm . TB and HF (Figs. 3D, H) had relatively large irregular filler particles and aggregated filler particles. CM (Fig. 3E) exhibited irregular filler particles and somewhat larger pre-polymerized filler particles.

Representative SEM images of the wear facets after simulated occlusal wear testing are shown in Figs. 4A-H. SEM images clearly showed that the morphological appearance of the wear facets were material and location dependent. Among the bulk-fill resin composites, SD and TB (Figs. 4C, D) showed rougher surfaces and larger facets as compared to those exhibited by the other bulk-fill resin composites. In addition, filler particles were clearly visible at the higher magnification. Although GB (Fig. 4B) showed a smaller and shallower wear facet than the other bulk-fill resin composites, the center of the facet showed some deep cleavages. FB (Fig. 4A) showed a smoother surface than the other bulk-fill resin composites. Among the flowable resin composites, the wear facet of GF (Fig. 4G) was similar to that of GB (Fig. 4B). Although the wear facets of CM, FF, and HF (Figs. 4E, F, and H) were smaller than those of bulk-fill resin composites, with the exception of GB, these surfaces were somewhat rough at the lower magnification. At the higher magnification, the filler particles of CM and FF were clearly visible. The wear pattern of GF (Fig. 4G) was similar to GB (Fig. 4B), that is, the wear facet was small and shallow, but deep cleavages were observed at the center of the facet.

Discussion

During occlusal contact, wear is caused by contact with opposing teeth or restorations, and is primarily considered attrition wear (12). The magnitude of the occlusal force is in the range of 10 to 20 N in the initial biting phase and increases to the range of 100 to 140 N in the molars and 25 to 45 N in the incisors at the end of the mastication cycle (13). This is why the force peak load for wear simulation should be in the range of 20 N to 150 N. Direct contacts

between antagonist teeth is about 15–20 min per day, depending on eating frequencies and habits. This does not include tooth contact during swallowing, which is typically of a lower magnitude. The mean chewing frequency ranges from 0.9 to 2.1 Hz with about 300 strokes per meal (14). If it is assumed that three meals are eaten per day, an individual carries out approximately 330,000 chewing cycles per year. In the present study, wear testing for resin composites was simulated using a peak force load of 78.5 N and frequency of 2 Hz over 400,000 cycles. These parameters were chosen to represent around 1 year of clinical function, and as a compromise between the forces experienced by incisors and molars.

The simulated occlusal wear of the resin composites evaluated in this study ranged from 0.025 to 0.148 mm³ for VL and from 98.1 to 210.6 μm for MD of bulk-fill and conventional flowable resin composites, and VL and MD were material dependent. GF, GB, and FF showed better wear resistance when compared to the other tested materials. Thus, the null hypothesis, that the simulated occlusal wear of bulk-fill and conventional flowable resin composites would not be influenced by the type of material, was rejected.

Tsujimoto *et al.* (15, 16) used the same wear apparatus to study the simulated occlusal wear of CAD/CAM resin composite blocks and some indirect resin composites using 400,000 cycles, and reported that wear ranged from 0.019 to 0.035 mm³ for VL and from 69.2 to 133.7 μm for MD. Therefore, GF, GB, and FF showed a similar level of wear resistance to CAD/CAM resin composite blocks and some indirect resin composites. These resin blocks are produced by factory polymerization, and indirect resin composites experience post-cure heat treatment in addition to photo polymerization. This is generally believed to create much harder resin composite restorations than can be obtained with direct application, and thus the comparable values obtained for some flowable resin composites in this experiment were unexpected.

It was speculated that the lower occlusal wear of GB and GF was attributed to the smaller particle size of fillers. From the manufacturer's information, homogeneously and densely dispersed ultra-fine 150 nm barium fillers were used in both GB and GF. These filler particles are much smaller than those in the other tested resin composites. It has been found that the filler particle size affects the friction coefficient and surface roughness, that act as determinants of the wear resistance of resin composites (17). Smaller filler particles might be associated with lower friction coefficients and resulted in lower internal shear stress in the polymer matrix. In addition, such improved wear resistance of flowable resin composites has been hypothesized to result from smaller interparticle spacing between the fillers of small-particle composites in occlusal contact-free areas. The smaller filler particles become more closely packed, so that the resin between them may be protected from further abrasion from neighboring particles (5). The same effect is seen in both conventional and bulk-fill flowable resin composites using the same sort of composition, and this suggested that bulk-fill flowable resin composites could be generally improved to match the quality of other available options.

The simulated occlusal VL of the resin composites evaluated ranged from 0.026 to 0.148 mm³ for bulk-fill flowable resin composites and 0.025 to 0.080 mm³ for conventional flowable resin composites. The simulated occlusal MD wear of bulk-fill flowable resin composites ranged from 103.8 to 210.6 μm and that of the conventional flowable resin composites evaluated ranged from 98.1 to 150.9 μm. Thus, based on the results of this study, the wear resistance of bulk-fill resin composites appears to be lower than those of conventional flowable resin composites. This is also true when comparing FF and FB, which have very similar compositions. This result is consistent with a previous study (18), which evaluated the physico-mechanical characteristics of bulk-fill resin composites compared to conventional resin composites.

The clinical implication of this study is that selected flowable resin composites can be used in occlusal contact areas in posterior restorations, and that flowable resin composites can be recommended for use in restorations which may be exposed to occlusal contact. However, because of the wide range of wear characteristic found in certain bulk-fill flowable resin composites, clinicians need to be careful when selecting a resin composite to use.

Conclusions

The results of this study showed that the simulated occlusal wear rates of GF, GB and FF were lower than those of the other tested resin composites, and the wear patterns were material dependent. The wear resistance of bulk-fill resin composites appeared to show an extremely broad range, much wider than that of the conventional flowable resin composites. Some resin composites of each type might be suitable for use in occlusal contact areas of posterior restorations, but care should be taken when selecting materials.

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Tables and Figures

Table 1 Materials used in this study

| Code | Material | Type of resin composite | Manufacturer |
|------|---------------------------------------|-------------------------|--|
| FB | Filtek Bulk Fill Flowable Restorative | Bulk-fill | 3M Oral Care, St. Paul, MN, USA |
| GB | G-ænial Bulk Injectable | Bulk-fill | GC, Tokyo, Japan |
| SD | SDR Flow+ | Bulk-fill | Dentsply Sirona, Milford, CT, USA |
| TB | Tetric EvoFlow Bulk Fill | Bulk-fill | Ivoclar Vivadent, Schaan, Lichtenstein |
| CM | Clearfil Majesty IC | Conventional flowable | Kuraray Noritake Dental, Tokyo, Japan |
| FF | Filtek Supreme Ultra Flow | Conventional flowable | 3M Oral Care |
| GF | G-ænial Universal Flow | Conventional flowable | GC |
| HF | Herculite XRV Ultra Flow | Conventional flowable | Kerr, Orange, CA, USA |

Table 2 Simulated wear measurements for resin composites

| Code | Type of resin composite | Volume loss (mm ³) | Maximum depth (µm) |
|------|-------------------------|--------------------------------|----------------------------|
| GF | Conventional flowable | 0.025 (0.005) ^a | 98.1 (20.5) ^a |
| GB | Bulk-fill | 0.026 (0.007) ^a | 103.8 (20.2) ^a |
| FF | Conventional flowable | 0.040 (0.009) ^a | 116.8 (19.1) ^{ab} |
| FB | Bulk-fill | 0.062 (0.014) ^b | 124.1 (23.1) ^b |
| CM | Conventional flowable | 0.067 (0.012) ^{bc} | 129.8 (25.9) ^{bc} |
| HF | Conventional flowable | 0.080 (0.009) ^c | 150.9 (12.2) ^c |
| TB | Bulk-fill | 0.127 (0.019) ^d | 205.3 (17.8) ^d |
| SD | Bulk-fill | 0.148 (0.033) ^c | 210.6 (27.8) ^d |

Same lower case letter in same vertical column indicates no significant differences at 5% significance level. Values in parentheses indicate standard deviation.

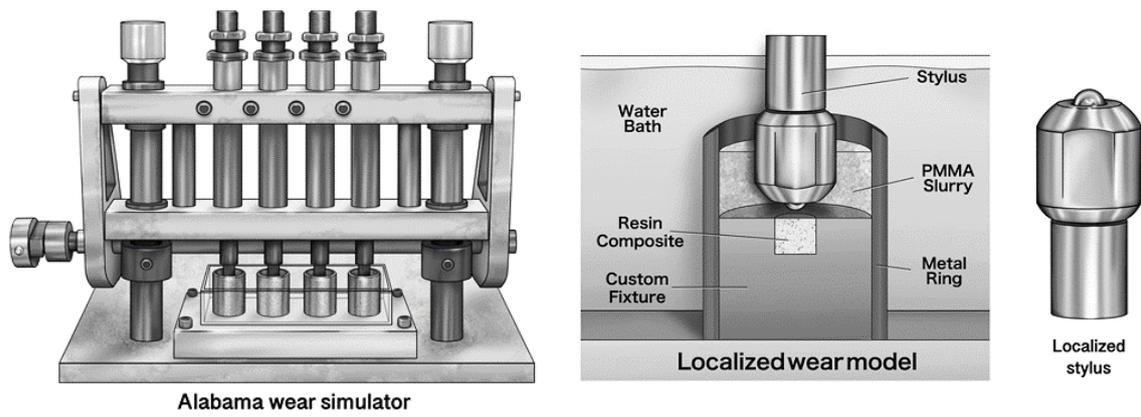


Fig. 1: Illustration of the set-up for occlusal wear simulation with the Alabama wear testing machine.

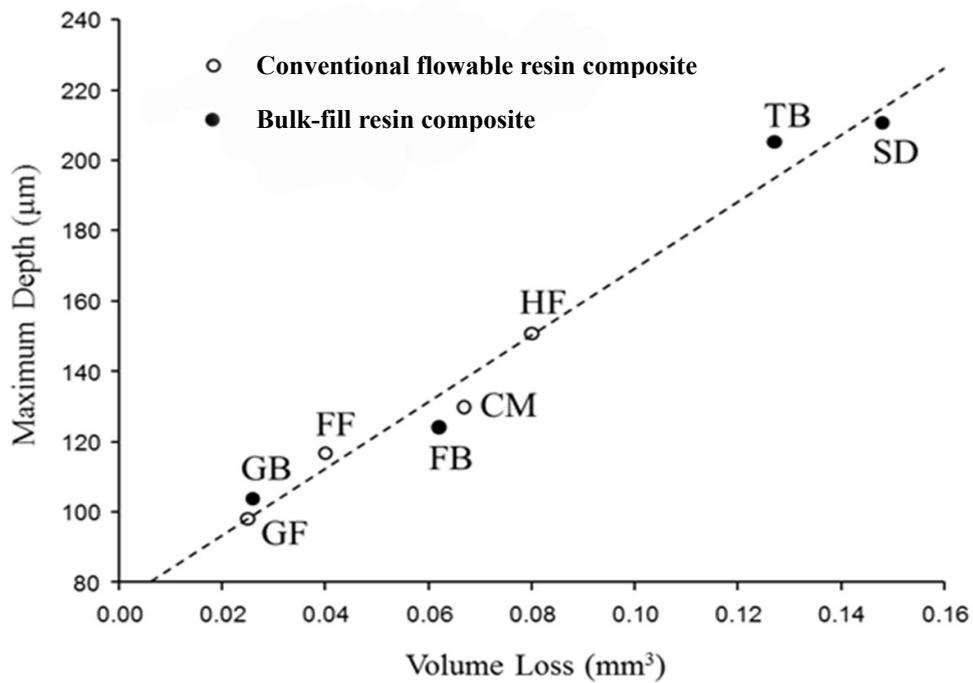


Fig. 2: Inter-relationship between VL and MD

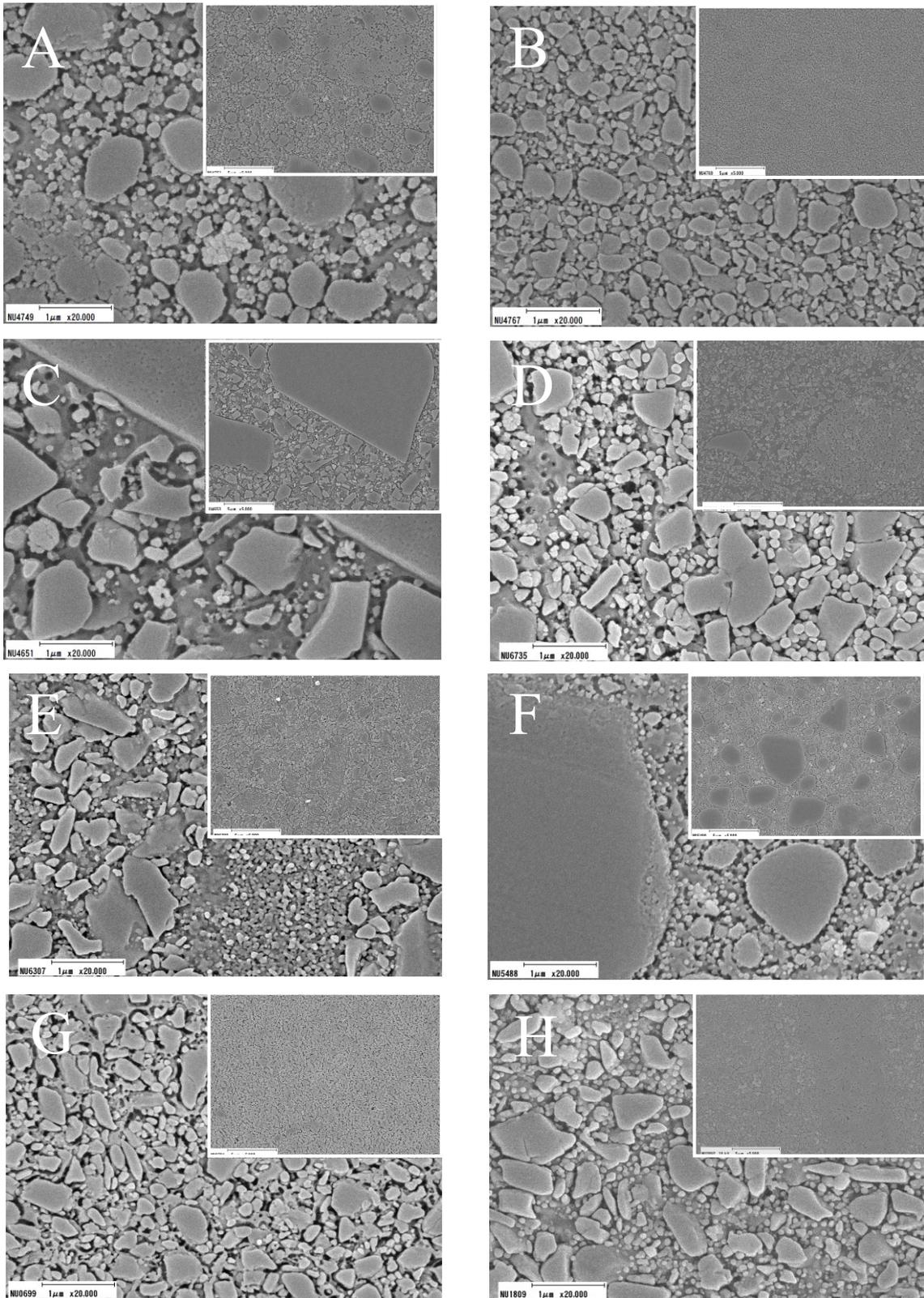


Fig. 3. SEM images of the argon-ion-etched surfaces of the bulk-fill and flowable resin composites. SEM images as viewed at magnifications 5,000 \times and 20,000 \times (A: FB, B: GB, C: SD, D: TB, E: CM, F: FF, G: GF, H: HF).

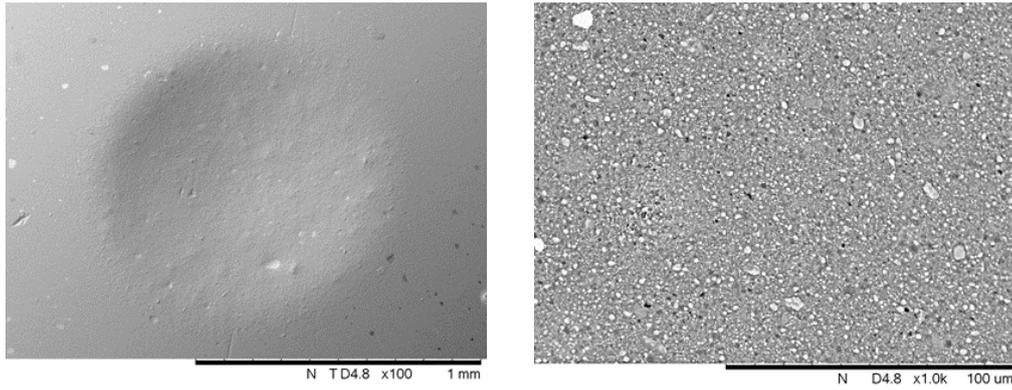


Fig.4A: SEM images of the wear facets of FB as viewed at 100× and 1,000×.

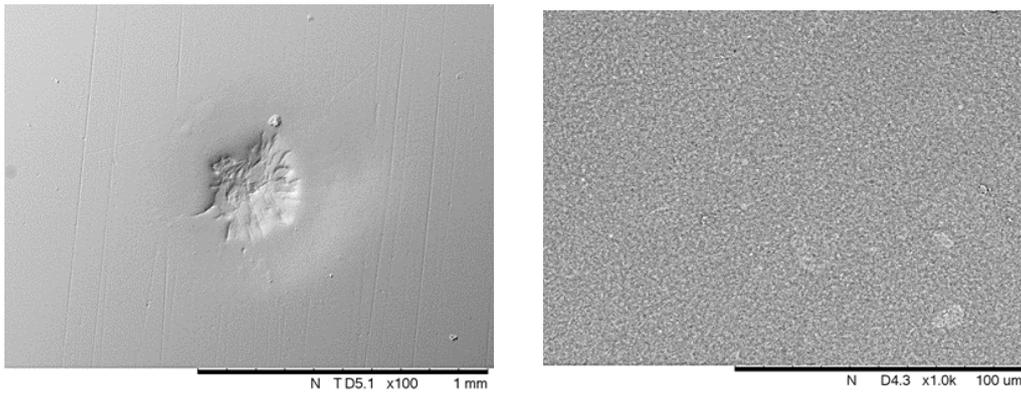


Fig. 4B: SEM images of the wear facets of GB as viewed at 100× and 1,000×.

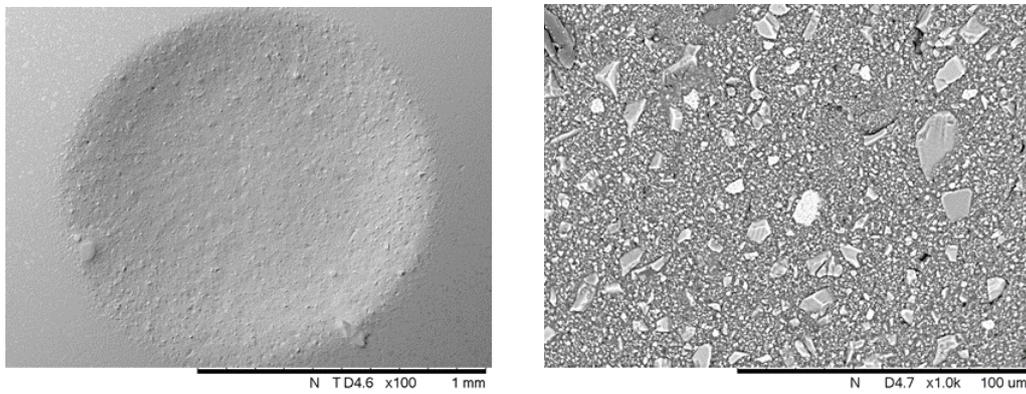


Fig. 4C: SEM images of the wear facets of SD as viewed at 100× and 1,000×.

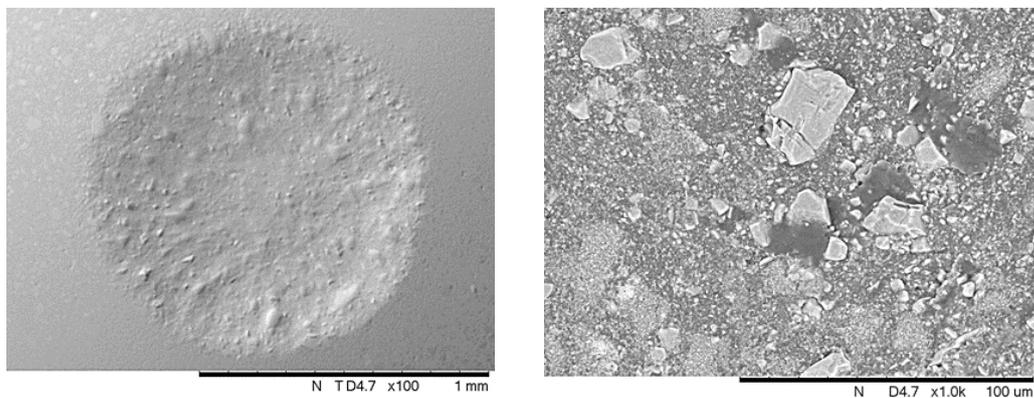


Fig. 4D: SEM images of the wear facets of TB as viewed at 100× and 1,000×.

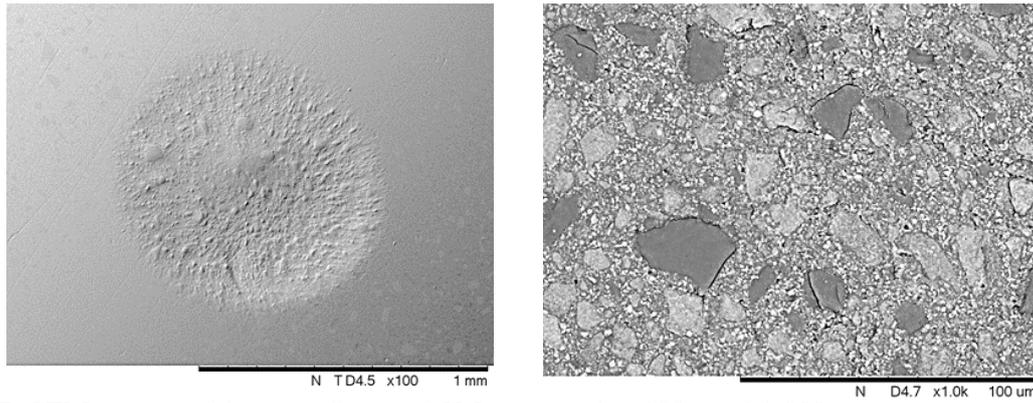


Fig. 4E: SEM images of the wear facets of CM as viewed at 100× and 1,000×.

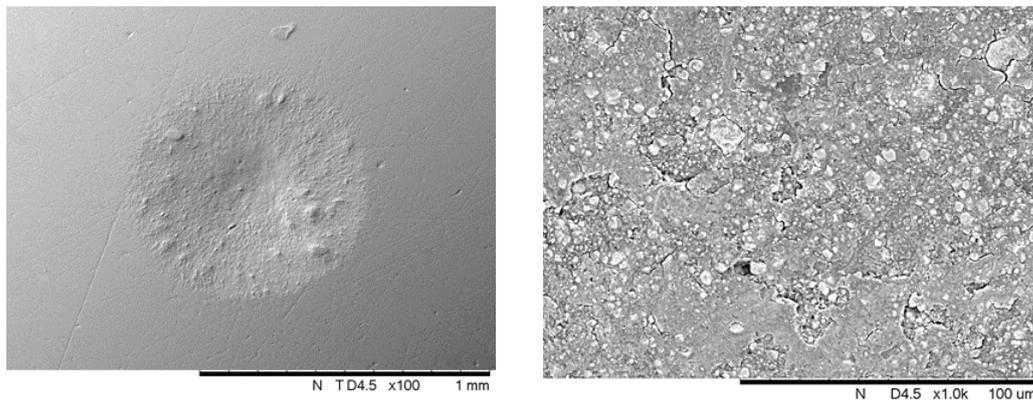


Fig. 4F: SEM images of the wear facets of FF as viewed at 100× and 1,000×.

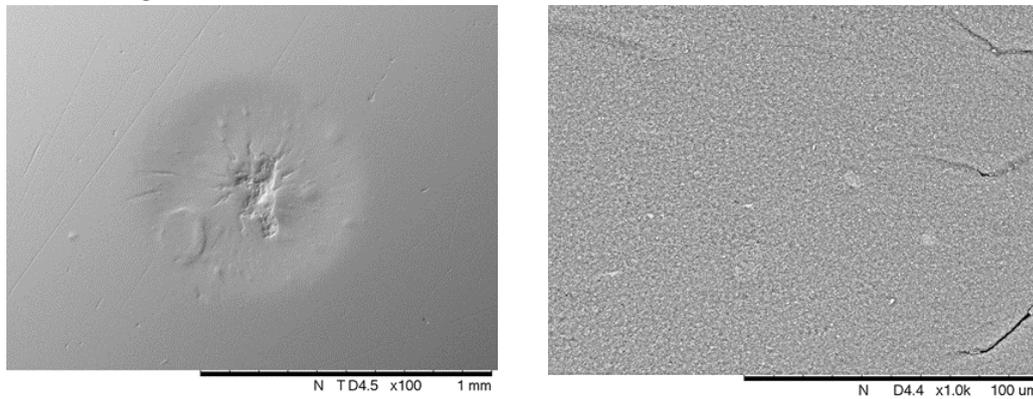


Fig. 4G: SEM images of the wear facets of GF as viewed at 100× and 1,000×.

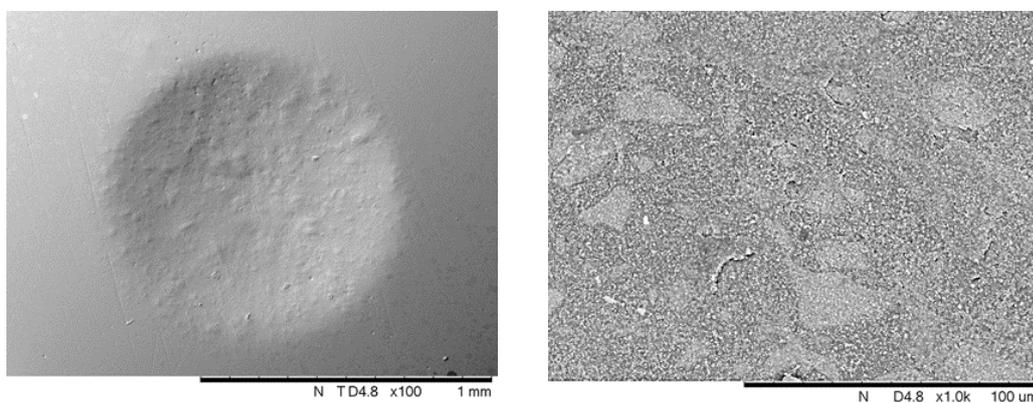


Fig. 4H: SEM images of the wear facets of HF as viewed at 100× and 1,000×.