Evaluation of the mechanical behavior of bulk-fill and conventional flowable resin composites using dynamic micro-indentation

Taro IWASAKI1, Naotaka KAMIYA2, Satoshi HIRAYAMA2 and Yasuhiro TANIMOTO1

1 Department of Dental Biomaterials, Nihon University School of Dentistry at Matsudo, 2-870-1 Sakae-cho Nishi, Matsudo, Chiba 271-8587, Japan
2 Department of Operative Dentistry, Nihon University School of Dentistry at Matsudo, 2-870-1 Sakae-cho Nishi, Matsudo, Chiba 271-8587, Japan

The purpose of this research was to investigate the mechanical behavior of commercially available bulk-fill and conventional flowable resin composites using the dynamic micro-indentation method. The effect of inorganic filler content on mechanical properties was also assessed. Weight percentages of the inorganic filler in the resin composite were measured using the ashing technique. The results showed that dynamic hardness and elastic modulus tended to increase with inorganic filler content. Furthermore, the differences in mechanical properties between top and bottom surfaces were less pronounced in bulk-fill flowable resin composites compared with conventional flowable resin composites. In conclusion, the mechanical properties of bulk-fill flowable resin composites are affected by filler content. Moreover, bulk-fill flowable resin composites have a higher polymerization depth than conventional flowable resin composites when sample thickness is 4 mm.

Keywords: Bulk-fill, Inorganic filler, Hardness test, Elastic modulus

INTRODUCTION

The physical properties and aesthetics of flowable resin composites can be improved by modifying the filler particles incorporated in the matrix, such as their concentration, size, and morphology. This has expanded their application to restorations in all areas of the oral cavity. However, the resin composites can undergo polymerization shrinkage, which causes clinical failure of the restoration, including secondary caries and discoloration.

To address the issue of polymerization shrinkage, an incremental technique is applied in restorative treatment. This technique not only addresses polymerization shrinkage, but ensures adequate light transmittance and resin composite polymerization. This is because the light-curing resin composites have a limited depth of cure. However, the incremental technique has disadvantages including void or contamination between material layers, adhesive failure between layers, placement difficulty in small cavities, as well as being time consuming for the dentist and inconvenient for the patient when dealing with large cavities.

Today, many of commercial bulk-fill resin composites are marketed with an increment depth of 4–5 mm. These products simplify the restorative procedure compared with the incremental technique, while also reducing polymerization shrinkage stress. Furthermore, applying bulk-fill flowable resin composites saves clinical time and prevents the formation of air bubbles and contamination between material layers.

The selection of filling materials to restore the cavity should be based on the mechanical property information of the bulk-fill flowable resin composite. There are several studies on the mechanical properties of bulk-fill flowable resin composites; however, dynamic hardness measured using a micro-indentation method has not been investigated. This method is widely used in other industries because of the easy specimen preparation involved, not only for industrial materials but also dental materials. Its application for investigating mechanical properties at small scales makes it particularly useful for restorative materials.

Additionally, the dynamic micro-indentation test has significant advantages over two commonly used methods (i.e., Vickers hardness and Knoop hardness). These surface hardness tests apply a fixed load on a diamond pyramidal indenter with a square base or a rhombic-based pyramidal diamond indenter. The area of the resulting indentation after unloading is then measured with an optical microscope; however, it is difficult to measure such dimensions on the micro-/nano-scale with a high degree of accuracy using this method. The dynamic micro-indentation method is a depth-sensing indentation hardness test that can continuously measure the load on, and the displacement of, a diamond Berkovich indenter tip until a given maximum load or depth is reached and then removed. This removes the need to observe indentations on the specimen surface with an optical microscope. The degree of total deformation including plastic deformation and elastic deformation can then be measured, which allows the mechanical parameters of dynamic hardness and elastic modulus to be computed. Hence, the dynamic micro-indentation method is an effective method for evaluating mechanical properties of bulk-fill flowable resin composites.

The purpose of this study was to examine the mechanical behavior of commercially available flowable resin composites using a dynamic micro-indentation tester and to evaluate the differences in mechanical properties between the top and bottom surfaces.
Moreover, the effects of inorganic filler content on the dynamic hardness and elastic modulus of bulk-fill flowable resin composites obtained from the indentation test are investigated.

**MATERIALS AND METHODS**

**Specimen preparation**

The commercially available resin composites investigated are listed in Table 1. Beautifil bulk flowable (BB; Shofu, Kyoto, Japan), Bulk base hard low flow (BH; Sun Medical, Moriyama, Japan), Filtek fill and core flowable restorative (FF; 3M, St. Paul, MN, USA), Gracefil bulkflo (GB; GC, Tokyo, Japan), and SDR (SD; Dentsply Sirona, Charlotte, NC, USA) are bulk-fill resin composites. Clearfil majesty ES flow low (CM; Kuraray Noritake Dental, Tokyo, Japan) and Estelite flow quick (EF; Tokuyama Dental, Tokyo, Japan) are conventional resin composites. A glass mold with a depth of 4 mm and inner diameter of 6 mm was used to prepare the specimens for the dynamic micro-indentation test. The layer thickness of specimens were applied in the depth of 4 mm according to the manufacturer’s instructions. The mold was placed on a glass slide filled with the

| Trade name and shade | Matrix | Fillers | Filler size (μm) | Filler shape | Manufacturer (Lot) |
|----------------------|--------|---------|-----------------|-------------|-------------------|
| **Bulk-fill resin composites** | | | | | |
| Beautifil bulk flowable | BB Universal | Bis-GMA, UDMA, Bis-MPEPP, TEGDMA | S-PRG\(^{15}\) based on fluoroboroaluminosilicate glass | NA | Irregular | Shofu, Kyoto, Japan (101936) |
| Bulk base hard low flow | BH Universal | Bis-MPEPP, urethane acrylate | Barium-silica-glass, strontium-silica-glass | 1, 3, 5 | Irregular | Sun Medical, Moriyama, Japan (TT11) |
| Filtek fill and core flowable restorative | FF Universal | Bis-GMA, UDMA, Bis-EMA, Procrylat resins | Zirconia/silica, Ytterbium trifluoride | 0.01–3.5, 0.1–5.0 | Irregular | 3M, St. Paul, MN, USA (NA64684) |
| Gracefil bulkflo | GB Universal | Bis-MEPP | Barium glass | 0.15 (average) | Irregular | GC, Tokyo, Japan (1908272) |
| SDR | SD Universal | Modified UDMA, Bis-EMA, TEGDMA | Barium-alumino-fluoroborosilicate glass, strontium alumino-fluorosilicate glass | 4.2 (average) | Irregular | Dentsply Sirona, Charlotte, NC, USA (1906000020) |
| **Conventional resin composites** | | | | | |
| Clearfil majesty ES flow low | CM A2 | TEGDMA, hydrophobic aromatic dimethacrylate | Silanated barium glass, silanated silica | 0.18–3.5 | Irregular | Kuraray Noritake Dental, Tokyo, Japan (5L0294) |
| Estelite flow quick | EF A2 | Bis-MPEPP, UDMA, TEGDMA | Silica-zirconia, silica-titania | 0.04–0.6 | Spherical | Tokuyama Dental, Tokyo, Japan (JO69) |

Bis-GMA: bisphenol A diglycidyl methacrylate, UDMA: urethane dimethacrylate, Bis-MPEPP: bisphenol A polyethoxy methacrylate, TEGDMA: triethyleneglycol dimethacrylate, Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate, Procrylat: 2,2-bis([1-(3-methacryloxypropoxy)phenyl]propane, Bis-MEPP: bisphenol A ethoxylate dimethacrylate, S-PRG: surface modified prereacted glass, NA: not available
composite material. The top of the composite was covered with polyester strips (Matrix tape, 3M) to remove excess material and inhibit contact with oxygen during light polymerization and then pressed using a glass slide. The specimen was light-polymerized through the strips using a laboratory light polymerization unit (α-Light V, J. Morita, Kyoto, Japan) according to the manufacturers’ instructions. The polymerization unit was light-emitting diode (LED)-based unit and equipped with blue-violet LED light sources. After polymerization, the strips were removed from the specimen and the top and bottom surface of the composite material were polished with 3000-grit silicon carbide paper (waterproof abrasive paper, Nihon Kenshi, Hiroshima, Japan) under running water to remove the oxygen-inhibiting surface layer. All specimens were stored in distilled water at 37ºC in the dark for 24 h.

Scanning electron microscopic observations
Representative surface images of the resin composite specimens were observed and captured by scanning electron microscope (SEM) at ×5,000 magnification (Fig. 1). Specimens were sputtered with gold-palladium alloy for 30 s by using an ion sputtering device (IB-5, EIKO, Tokyo, Japan) and observations were conducted with a SEM (S-3400N, Hitachi High-Technologies, Tokyo, Japan) operated at an accelerating voltage of 15 kV.

Dynamic micro-indentation test
The dynamic hardness of the top and bottom surfaces of each specimen was measured using a dynamic ultra-micro-hardness tester (DUH-211, Shimadzu, Kyoto, Japan) fitted with a Berkovich indenter tip. Specimens were affixed to an attached holder. Dynamic microindentation primarily comprises a controlled load (P) applied through a diamond tip, which is in contact with a smooth surface. The penetration depth (h) of the indentation is continuously recorded as a function of load. Figure 2 shows a schematic illustration of a typical indentation load-penetration depth curve acquired by a dynamic micro-indentation test.

During indenter loading and unloading, the specimen is subjected to both plastic deformation (h_p) and elastic deformation (h_e). The total deformation (h_t) is the sum of h_p and h_e in the micro-indentation load-penetration depth curve. Moreover, the dynamic hardness (DH) and elastic modulus (E) can be obtained from the indentation load and penetration depth data.

DH of the specimen is calculated from the following equation:

\[ DH = \frac{2P}{h^2} \]

![Fig. 1 SEM images of typical surfaces for the resin composites investigated here (original magnification ×5,000).](image1)

![Fig. 2 Schematic illustration of a typical micro-indentation versus penetration depth curve and flowchart of hardness testing.](image2)
where \( \alpha \) is a geometrical constant of the Berkovich indenter (3.8584), \( P \) is the applied load during the indentation test, and \( h \) is the penetration depth of indentation.

\[
E = \frac{1}{E_i} \left( \frac{1}{E} + \frac{1}{E_i} \right),
\]

where \( E_i \) is the reduced elastic modulus from the indenter, \( V \) is Poisson’s ratio for the specimen, \( V_i \) is Poisson’s ratio for the Berkovich indenter (0.07), and \( E \) is the elastic modulus of the Berkovich indenter (1,140 GPa)\(^{14,15} \).

The dynamic micro-indentation tests were performed with peak loads (\( P_{\text{max}} \)) of 196.1 mN. The load rate was kept constant at 13.32 mN/s, and the hold time at maximum load was set to 15 s. The dynamic hardness and elastic modulus on the top and bottom surface of specimen were obtained by averaging the values of six specimens, which were each measured three times.

### Weight percentage of inorganic fillers

The weight percentage of the inorganic fillers in the resin composites were measured using the ashing technique. A porcelain crucible containing approximately 0.2 g of the composite paste was sintered at a maximum temperature of 550ºC under atmospheric pressure in a furnace (MSFT-1520-P, Nikkato, Tokyo, Japan). The temperature was increased at a rate of 5ºC/min to the maximum temperature, and the hold time was set to 2 h at 550ºC. The weight percentages of the fillers were determined by calculating the difference in weight of the specimen before and after sintering using a precision balance (AG285, Mettler-Toledo, Greifensee, Switzerland). Three specimens of each material were analyzed.

### Statistical analysis

The dynamic hardness and elastic modulus results from the dynamic micro-indentation tests were analyzed using a two-way analysis of variance followed by a Tukey’s test for multiple comparisons of means. The Pearson correlation analysis was performed to evaluate the relationships between the inorganic filler content and the dynamic hardness and elastic modulus. All statistical analysis was performed using statistical software (BellCurve for Excel, Social Survey Research Information, Tokyo, Japan). The significance level was defined as \( p<0.05 \).

### RESULTS

The total deformation \( (h_t) \) values of the top and bottom surfaces of each specimen and the statistical analysis results are presented in Table 2. It can be seen that the total deformation equation, \( h_t = h_p + h_e \), was always satisfied. The plastic and elastic deformation rates \( (h_p/h_t \text{ and } h_e/h_t) \) are also reported. All specimens showed larger total, plastic, and elastic deformations on the bottom surface than the top. Both surfaces of BH showed significantly higher values in \( h_t \) and \( h_p \) than the other specimens \( (p<0.05) \) and also had the highest \( h_p/h_t \) ratio.

The results of the dynamic hardness and elastic moduli of the resin composites as well as the weight percentages of inorganic filler are summarized in Table 3. Inorganic filler content ranged from 61.2 to 69.5 wt% and BB had the highest filler content. The dynamic

### Table 2  Total, plastic, and elastic deformations (mean\(=\)standard deviation) of the resin composites with surfaces (top and bottom)

| ID | Surfaces | Total deformation, \(h_t\) (\(\mu m\)) | Plastic deformation, \(h_p\) (\(\mu m\)) | \(h_p/h_t\) (%) | Elastic deformation, \(h_e\) (\(\mu m\)) | \(h_e/h_t\) (%) |
|----|---------|----------------------------------------|----------------------------------------|----------------|----------------------------------------|----------------|
| BB | Top     | 4.8±0.074\(^a\)                       | 2.1±0.077\(^a\)                       | 43.9           | 2.7±0.11\(^a\)                       | 56.1           |
| BH | Top     | 7.0±0.25\(^b\)                        | 3.7±0.31\(^b\)                       | 52.6           | 3.3±0.22\(^bc\)                      | 47.4           |
| FF | Top     | 6.0±0.15\(^c\)                        | 2.8±0.021\(^c\)                      | 47.4           | 3.1±0.13\(^b\)                      | 52.6           |
| GB | Top     | 5.7±0.24\(^d\) \(\pm\) 2.2±0.073\(^a\) | 52.3           | 3.6±0.15\(^e\) | 47.7           | 52.6 |
| CM | Top     | 5.2±0.096\(^a\)                       | 2.0±0.064\(^d\) \(\pm\) 37.7         | 3.2±0.067\(^b\) | 62.3           |
| EF | Top     | 4.4±0.073\(^d\)                       | 1.8±0.040\(^d\) \(\pm\) 40.5         | 2.6±0.093\(^d\) | 59.5           |
| BB | Bottom  | 5.7±0.34\(^a\)                        | 2.3±0.27\(^a\)                       | 41.0           | 3.3±0.38\(^b\)                      | 59.0           |
| BH | Bottom  | 8.1±0.28\(^b\)                        | 4.5±0.43\(^b\)                       | 56.1           | 3.6±0.23\(^b\)                      | 43.9           |
| FF | Bottom  | 7.3±0.12\(^c\)                        | 3.7±0.13\(^c\)                       | 51.3           | 3.6±0.24\(^e\)                      | 48.7           |
| GB | Bottom  | 6.7±0.34\(^d\) \(\pm\) 2.9±0.23\(^d\) | 44.1           | 3.7±0.15\(^e\) | 55.9           |
| SD | Bottom  | 6.2±0.23\(^c\)                        | 3.4±0.19\(^c\)                       | 55.5           | 2.7±0.072\(^c\)                      | 44.5           |
| CM | Bottom  | 6.7±0.082\(^d\) \(\pm\) 2.8±0.095\(^d\) | 41.3           | 4.0±0.061\(^d\) | 58.7           |
| EF | Bottom  | 5.8±0.21\(^ax\) \(\pm\) 2.7±0.16\(^ax\) | 47.2           | 3.1±0.23\(^cx\) | 52.8           |

The same superscript characters (a–f) indicate no significant difference from each other \( (p>0.05) \).
Table 3  Means of dynamic hardness and elastic moduli (mean±standard deviation) of the resin composites with surfaces (top and bottom)

| ID | Inorganic filler content (wt%) | Dynamic hardness (-) | Elastic modulus (GPa) |
|----|-------------------------------|----------------------|-----------------------|
|    |                               | Top                  | Bottom                | Top     | Bottom       | Bottom/top ratio |
| BB | 69.5                          | 33.0±1.0 ±2.8         | 0.729                 | 9.5±0.9±b| 6.6±0.7±a  | 0.700          |
| BH | 68.7                          | 15.9±1.2±2.3±3.3      | 0.737                 | 6.0±0.4±d| 5.1±0.2±c  | 0.849          |
| FF | 61.3                          | 21.6±1.1±1.3±2.5      | 0.667                 | 6.8±0.7±c| 5.0±0.7±c  | 0.734          |
| GB | 61.2                          | 23.7±1.8±3.3±4.3      | 0.727                 | 5.5±0.4±d| 4.9±0.4±c  | 0.879          |
| SD | 66.3                          | 25.6±2.0±3.2±4.3      | 0.788                 | 8.5±1.0±d| 6.8±0.7±c  | 0.792          |
| CM | 63.7                          | 28.1±1.0±1.3±2.4      | 0.598                 | 7.0±0.3±c| 4.6±0.3±c  | 0.648          |
| EF | 68.6                          | 40.1±1.3±3.3±4.5      | 0.571                 | 9.9±0.7±b| 6.4±0.6±c  | 0.650          |

The same superscript characters (a–f) at each vertical column indicate no significant difference from each other and the same superscript character (A) at horizontal line indicates no significant difference from each other (p>0.05).

The hardness of the resin composites ranged from 15.9 to 40.1 for the top surface and 11.7 to 24.0 for the bottom. The bottom/top dynamic hardness ratio of BB, BH, FF, GB, and SD were higher than CM and EF. The elastic moduli of the resin composites ranged from 5.5 to 9.9 for...
Fig. 5 Relationship between the inorganic filler content and the elastic modulus for resin composites. 

- Top: \( y = 0.313x - 12.917 \) \( R^2 = 0.413 \)
- Bottom: \( y = 0.174x - 5.793 \) \( R^2 = 0.437 \)

The inorganic filler content obtained from this analysis was less than the manufacturers' value for each specimen where a value was provided. The first reason for this is the amount of silane. Generally, resin composite is reinforced by the dispersion of silica treated with a silane coupling agent (e.g., \( \gamma \)-methacryloxypropyltrimethoxysilane) and has superior mechanical properties because of a siloxane bond established between the silane-treated silica filler and organic matrix. It seems that manufacturers include the weight of the silane coating when calculating the weight percentage of fillers. Accordingly, it is likely that the large surface area of fillers, caused by nano-sized particles and porous clusters, result in a large quantity of silane captured by the manufacturers but not in this analysis.

For BH, the total deformation and \( h_p/h_t \) ratio was higher than the other resin composites despite having a higher inorganic filler content. Additionally, the dynamic hardness and elastic modulus of BH were significantly lower compared with the other groups. Nitta et al.\(^{32}\) reported that the reason for the poor mechanical properties of BH can be insufficient chemical bonding between fillers and the matrix by silane treatment. They found the failure of fillers in BH after polishing with silicon carbide paper from the SEM observation. That is, filler failures cause the poor mechanical properties.
which supports our findings on the disadvantages of BH.

Figures 4 and 5 indicate that there is no linear correlation between the inorganic filler content and dynamic hardness or elastic modulus for BH. However, when performing the correlation analysis on the other composites, there was a significant correlation for both the top and bottom surface. The correlation between the inorganic filler content and dynamic hardness had $R^2$ values of 0.678 ($p=0.044$) for the top surface and 0.910 ($p=0.003$) for the bottom surface. The correlation between the inorganic filler content and the elastic modulus had $R^2$ values of 0.914 ($p=0.003$) for the top surface and 0.725 ($p=0.032$) for the bottom surface. The filler content was significantly correlated to the mechanical properties of the resin composites as reported in other studies.$^{24,28}$

In the present study, the differences in mechanical properties such as dynamic hardness and elastic modulus between top and bottom surfaces were defined as the bottom/top ratio. To be brief, the higher the bottom/top ratio is, the higher the polymerization depth. The bottom/top ratio for dynamic hardness and elastic moduli were lower in conventional flowable resin composites than the bulk-fill flowable resin composites. Based on these results, it follows that the bulk-fill flowable resin composites have better properties at a higher depth of polymerization when the specimen thickness is 4 mm. Conversely, conventional flowable resin composites are designed to be placed in thinner single increments (up to 2 mm) compared with bulk-fill flowable resin composites (up to 4 mm). The recommended incremental depth is assumed to affect the bottom/top ratio.

The term “creep” refers to the time-dependent change in deformation under constant stress$^{20}$ and is attributed to viscoelastic properties. In the indentation load-penetration depth curves, it is clearly observed that the indentation creep on bottom surfaces are larger than the top surfaces (see Fig. 3). As clinicians restore cavities using resin composites in clinical situations, the difference in creep because of layer thickness can cause clinical problems and decrease clinical performance. Singh et al.$^{34}$ pointed out that a large degree of creep in resin composites used to fill in a cavity will lead to the failure of the restoration and pulp inflammation from debonding and fracture.

Furthermore, judging from $h_t/h_b$ and $h_b/h_t$ ratio results, the mechanical properties of each resin composite differs depending on the material types and their surfaces. Ferracane and Greener$^{26}$ suggest that mechanical properties of restorative dental resins were affected by the degree of conversion of carbon-carbon double bonds (C=C) brought about by the quality of the cross-linked network formed in the resin. There are also previous studies that increase filler content to reduce creep strain$^{30}$ and found that creep depends on the degree of photo polymerization of resin composites.$^{37}$ Therefore, additional investigations should be conducted to determine all factors that affect mechanical properties and to ascertain the relationship between the creep and the composition of resin composites, such as the matrix, filler content or size, and the degree of conversion.

All in vitro studies have limitations. Factors known to affect the long-term clinical outcomes of resin composite restorations, for example, constituents, monomer conversion, polymerization shrinkage, and 3-dimensional cavity design were not assessed in this study. Clinical trials are necessary to verify the final evaluation of the materials used in the present study.

CONCLUSIONS

This study investigated the mechanical behavior of bulk-fill flowable resin composite using the dynamic micro-indentation method. The following conclusions were drawn.

1. In all flowable resin composites tested except for BH, a linear correlation was identified between inorganic filler content of resin composites and the mechanical properties (the dynamic hardness and elastic modulus) measured by the dynamic micro-indentation test.

2. Bulk-fill flowable resin composites had higher bottom/top ratio of dynamic hardness and elastic moduli than conventional flowable resin composites; bulk-fill flowable resin composites had the higher depth of polymerization when the specimen thickness is 4 mm. It was concluded that bulk-fill flowable resin composites were designed to be placed in deeper increments compared with conventional flowable resin composites.

3. There were differences in indentation creep deformations between surfaces and the indentation depth was larger in the bottom surface than the top.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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