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Physical properties and wear behavior of CAD/CAM resin composite blocks containing S-PRG filler for restoring primary molar teeth

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ABSTRACT

Objectives: This study aimed to develop computer-aided design/computer-aided manufacturing (CAD/CAM) resin composite blocks (RCBs) containing surface pre-reacted glass-ionomer (S-PRG) filler for primary molar teeth and evaluate their physical properties and wear resistance.

Methods: Experimental CAD/CAM RCBs containing S-PRG filler for primary molar teeth (EB), a commercial CAD/CAM RCB (HC), two resin composites for primary teeth (BKP and BKZ) and one for permanent teeth (BII) were used. Hardness tests, three-point bending tests, fracture toughness tests, and water absorption tests were conducted. Wear tests were conducted for these materials and stainless steel crowns (SSCs).

Results: The Vickers hardness of EB was lower than that of HC ($p < 0.05$), and there was no significant difference among BKZ, BKP, and BII ($p > 0.05$). After 1 week of water immersion, EB and HC showed greater flexural strength than the other materials ($p < 0.05$). EB showed greater fracture toughness than the other materials ($p < 0.05$). The water absorption of EB was lower than that of HC, BKZ, and BKP ($p < 0.05$), and greater than that of BII ($p < 0.05$). Antagonist wear was significantly smaller in EB than in HC and BII ($p < 0.05$), and significantly greater than in BKZ ($p < 0.05$). Antagonist wear could not be measured in SSC because of excessive wear that was out of range of the surface roughness tester.

Significance: The CAD/CAM RCBs containing S-PRG filler for primary molar teeth developed in this study demonstrated adequate physical properties and wear performance, suggesting that they are suitable for restoration of primary molar teeth and could function in place of SSCs.

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1. Introduction

In pediatric dentistry, ready-made stainless steel crowns (SSCs) have been widely used as effective restorations since the 1950s because of their high strength and long-term durability [1]. Tooth structure weakened by excessive preparation can be restored with a SSC with a high success rate [2]. However, SSC restorations have a disadvantage in terms of their non-esthetic appearance [3], and some parents and children prefer to avoid the use of SSCs for their children. Therefore, a new esthetic crown material for primary posterior teeth is required. Prefabricated pediatric zirconia crowns are one esthetically pleasing alternative for restoring primary posterior teeth [4]. However, prefabricated pediatric zirconia crowns [5] are harder than conventional SSCs [6] and could lead to wear of the antagonist teeth [7].

Computer-aided design/computer-aided manufacturing (CAD/CAM) resin composite crowns are available for use in the restoration of permanent posterior teeth because they are pre-polymerized under standardized industrial conditions, which improves their physical properties and wear resistance [8]. Antagonist enamel wear is reported to be lower against CAD/CAM resin composite crowns than against ceramic crowns [9]. CAD/CAM resin composite crowns could be an alternative restoration to prefabricated pediatric zirconia crowns. Thus, we started to develop a new CAD/CAM resin composite block (RCB) for primary molar teeth.

Surface pre-reacted glass-ionomer (S-PRG) fillers have been well established as an innovative technology of great interest, given their characteristic of releasing multiple ions, including fluoride (F^-), aluminum (Al^{3+}), borate (BO_3^{3-}), sodium (Na^+), silicate (SiO_3^{2-}), and strontium (Sr^{2+}) [10]. Resin composites containing S-PRG filler inhibit the growth of *Streptococcus mutans* on their surface by releasing BO_3^{3-} and F^- ions [11,12]. Moreover, the eluate from S-PRG fillers effectively inhibits *S. mutans* growth through downregulation of operons related to the sugar metabolism of *S. mutans*, thus reducing its cariogenicity [13]. These bio-functional abilities inhibit plaque accumulation. It has also been reported that the release of F^- and SiO_3^{2-} from resins containing S-PRG fillers promotes apatite formation [14], and F^- improves the acid resistance of the tooth substrate through the formation of fluoroapatite. Additionally, Sr released from S-PRG filler could be incorporated into the Ca site of hydroxyapatite, improving the acid resistance and remineralization [15]. The findings of previous studies indicate that the inclusion of S-PRG fillers to CAD/CAM RCBs has the capacity to prevent secondary caries as a result of an antibacterial effect and strengthening of the tooth structure caused by multiple-ion release. CAD/CAM resin composites combined with S-PRG technology can provide a suitable restoration for primary molar teeth with adequate physical properties and additional bio-functional ability.

The aim of this study was to develop CAD/CAM RCBs containing S-PRG filler for primary molar teeth and evaluate their physical properties and wear resistance. The null hypotheses examined were: (i) that CAD/CAM RCBs containing S-PRG filler do not possess adequate physical properties for the restoration of primary molar teeth when compared with conventional CAD/CAM RCBs and resin composites for primary and permanent teeth; and (ii) that CAD/CAM RCBs containing S-PRG filler do not cause less antagonist enamel wear than these conventional materials and stainless steel.

2. Materials and methods

2.1. *Preparation of specimens*

Experimental CAD/CAM RCBs containing S-PRG filler for primary molar teeth (EB; Shofu, Kyoto, Japan) were prepared. A commercial CAD/CAM RCB (Shofu Block HC, HC; Shofu), two resin composites for primary teeth (BEAUTIFIL Kids Paste, BKP and Zero flow, BKZ; Shofu) and one for permanent teeth (BEAUTIFIL II, BII; Shofu) were also used. Detailed compositions of each material are summarized in Table 1.

2.2. *Hardness test*

CAD/CAM RCBs were cut using a low-speed saw (Isomet, Buehler, Tokyo, Japan) under water irrigation into $14.0 \times 12.0 \times 1.0$ mm bars (n = 10). For the resin composites, a stainless steel mold (ϕ14.0 × 1.0 mm) was used to shape the specimens. The mold was filled with resin composite paste and a Mylar strip was applied. Light curing was conducted with an irradiance of 2000 mW/cm^2 (Pencure 2000; Morita, Kyoto, Japan). All specimens were polished with 2000-grit silicon carbide (SiC) paper and stored in distilled water at 37 °C for 1 week. The Vickers hardness was measured using a hardness testing machine (HM-211; Mitutoyo, Kanagawa, Japan) with 200 g loading for 8 s and a 10-second dwell time.

2.3. *Three-point bending test*

For all materials, $4.0 \times 1.2 \times 14.0$ mm bars were prepared (n = 10). The light curing and polishing process was the same as that for the Vickers hardness test. A three-point bending test was conducted using a universal testing machine (EZ-SX; Shimazu, Kyoto, Japan) with a 1.0 mm/min crosshead speed.

Table 1 – Composition of computer-aided design/computer aided manufacturing (CAD/CAM) resin composites and other resin composites used for fillings. Bis-GMA: Bisphenol A-diglycidyl methacrylate, Bis MPEPP: 2,2-bis(4-methacryloxypolyethoxyphenyl)propane, TEGDMA: Triethyleneglycol dimethacrylate, UDMA: Urethane dimethacrylate, UDA: Urethane diacrylate.

Materials	Abbreviation	Filler	Monomer	Filler content (wt%/vol%)	Average particle size of filler (μm)
Experimental CAD/CAM resin composite block	EB	S-PRG filler Multi-functional glass filler Ultra-fine filler	Bis-GMA, Bis MPEPP TEGDMA	63.4/41.7	0.4
Shofu Block HC; Shofu	HC	Silica filler Zirconium silicate Micro fumed silica	UDMA TEGDMA	68.0/52.0	2 ~ 15 1 ~ 10 0.01 ~ 0.04
BEAUTYFIL Kids Zero flow; Shofu	BKZ	S-PRG filler Multi-functional glass filler Ultra-fine filler	Bis-GMA TEGDMA	67.3/43.7	0.8
BEAUTYFIL Kids Paste; Shofu	BKP	S-PRG filler Multi-functional glass filler Prepolymerized filler Ultra-fine filler	Bis-GMA, TEGDMA UDA	82.6/69.3	Inorganic filler: 0.8 Prepolymerized filler: 25
BEAUTYFIL II; Shofu	BII	S-PRG filler Multi-functional glass filler Ultra-fine filler	Bis-GMA, TEGDMA UDA	83.3/67.7	0.8

Flexural strength, σ_m, was calculated using the following formula:

$$\sigma_m = \frac{3Pl}{2wb^2},$$

where P is the load at fracture, l the span, w the specimen width, and b the specimen height.

2.4. Fracture toughness test

For all materials, 6 × 6 × 6 × 12 mm triangular prisms were prepared (n = 10). CAD/CAM RCBs were cut using a low-speed saw (Isomet; Buehler) under water irrigation. A cutting machine (DWX-51; Roland DG, Shizuoka, Japan), CAM software (GO2dental Ver.6.02; Shofu), and a milling bar (CAD/CAM milling bar ball and diamond coating ϕ2.0 and ϕ1.0; Shofu) were used. For resin composites, stainless steel molds (6 × 6 × 6 × 12 mm) were used. The molds were filled with resin composite paste, which was light cured with an irradiance of 2000 mW/cm^2. All specimens were polished with 2000-grit silicon carbide (SiC) paper and stored in distilled water at 37 °C for 1 week. Fracture toughness was measured using a universal testing machine (EZ-SX; Shimazu) with a 1.0 mm/min crosshead speed. Fracture toughness, K_{Ic}, was calculated using the following formula:

$$K_{Ic} = \frac{P}{D\sqrt{W}} Y_{min},$$

where P is the load at fracture, D is the length of the prism, W is the length of the mold, and Y_{min} is the stress intensity factor. To obtain the fracture toughness, a notchless triangular prism test was conducted [16].

2.5. Water absorption test

For all materials, ϕ14.0 × 1.0 mm samples were prepared (n = 10). The light curing and polishing process were the same as that used for the Vickers hardness test. After polishing with 2000-grit SiC paper, the specimens were stored in the desiccator for drying at 37 °C for the first 22 h and at 22 °C for the next 2 h. The mass of the specimens was measured with an electronic scale (ER-60A; Kensei, Tokyo, Japan) and the cycle was repeated until the mass reached a constant weight. The specimens were stored in distilled water at 37 °C for 1 week. After drying, the mass of the specimens' absorbed water was measured and the drying cycle was repeated. The amount of water absorption, W_{sp}, was calculated using the following formula:

$$W_{sp} = \frac{m_2 - m_1}{V},$$

where V is the volume of specimens, m_1 is the mass of specimens stored in the water for 1 week, and m_2 is the mass of dried specimens after storing in the water for 1 week.

2.6. Wear test

First and second primary molars (n = 8, one each of left and right, upper and lower) were used as the antagonists. They were collected under the approval of the Institutional Review Board of Osaka University Dental Hospital (IRB No. H29-E28). The primary teeth were embedded in stainless steel molds ($\phi 16 \times 15$ mm) using an epoxy resin (EpoxiCure 2; Buehler). A small area ($3 \times 3\, mm^2$) of the surface of the enamel was exposed by polishing the mold. Severely worn or broken teeth and teeth with caries were excluded. Specimens and antagonists were mounted on a chewing simulator (K613; Tokyo Giken, Tokyo, Japan). For each specimen, a stylus with a 3 mm radius was prepared. As a control, a stainless steel (SUS304, Trueseed, Kyoto, Japan) stylus was also prepared. The specimens were tested using a vertical load of 75 N and a 30° clockwise rotation for 20,000 cycles under distilled water at 37 ℃ [17]. After the wear test, the depth of wear (means and standard deviations [SDs]) of the antagonist teeth and the tested materials was measured with a surface roughness (R_z) tester (Surftest SJ-400; Mitutoyo). The surface of each material after the wear test was observed under scanning electron microscopy (SEM) (TM3000, Hitachi High-Tech, Tokyo, Japan) and three-dimensional microscopy (VR-3200, Keyence, Osaka, Japan).

2.7. Statistical analysis

All results (except for flexural strength) were analyzed with one-way analysis of variance (ANOVA) ($\alpha = 0.05$) using SPSS Statistics (Version 21; IBM; Armonk, NY, USA). Post hoc analyses were conducted using the Dunnet T3 test/Tukey HSD test. The results for flexural strength were analyzed using two-way ANOVA ($\alpha = 0.05$). Post hoc analyses were conducted using the Bonferroni test ($\alpha = 0.05$).

3. Results

3.1. Vickers hardness of CAD/CAM resin composites, and resin composites for primary teeth and permanent teeth

Fig. 1a shows the Vickers hardness of the five specimens. EB recorded a lower Vickers hardness than HC ($p < 0.05$), and there was no significant difference among BKZ, BKP, and BII ($p > 0.05$).

3.2. Flexural strength of CAD/CAM resin composites, and resin composites for primary teeth and permanent teeth

Fig. 1b shows the flexural strength of the five specimens. In all materials except BII, the flexural strength was significantly lower after immersion for 1 week than after immersion for 24 h ($p < 0.05$). After water immersion for 1 week, there was no significant difference in flexural strength between EB and HC ($p > 0.05$), and these materials recorded greater flexural strength than the other materials ($p < 0.05$).

3.3. Fracture toughness of CAD/CAM resin composites, and resin composites for primary teeth and permanent teeth

Fig. 1c shows the fracture toughness of the five specimens. EB recorded a greater fracture toughness than the other materials ($p < 0.05$).

3.4. Water absorption of CAD/CAM resin composites, and resin composites for primary teeth and permanent teeth

Fig. 2 shows the amount of water absorption of the five specimens. EB recorded less water absorption than HC, BKZ, and BKP ($p < 0.05$), and more water absorption than BII ($p < 0.05$).

3.5. Wear of CAD/CAM resin composites, resin composites for primary teeth and permanent teeth, and stainless steel

Fig. 3a shows the amount of wear for the five specimens and the antagonist enamel. EB showed significantly smaller antagonist wear than HC and BII ($p < 0.05$), and significantly greater antagonist wear than BKZ ($p < 0.05$). Except for BII, there were no significant differences in the wear of the five materials ($p > 0.05$). The antagonist wear of SSC could not be measured because of excessive wear that was out of the range of the surface roughness tester. The amount of wear of SSC was not significantly different from that of EB. By SEM observation, a smooth surface was confirmed for EB and BKZ, while HC exhibited some cracks and a rough surface. Dentinal tubules were exposed and crack lines were confirmed at the center of the EB specimen.Fig. 4.

4. Discussion

The null hypotheses were rejected because the CAD/CAM RCBs containing S-PRG filler for primary molar teeth developed in this study demonstrated adequate physical properties and wear resistance to function as restorations for primary molar teeth. We believe that the CAD/CAM RCBs are suitable for fabrication of full crowns to restore primary teeth and have potential as an alternative material for conventional SSCs. Fig. 5.

The Vickers hardness of EB obtained in this study was lower than that of human enamel [18] and lower than that of HC. In a previous study, a positive correlation was reported

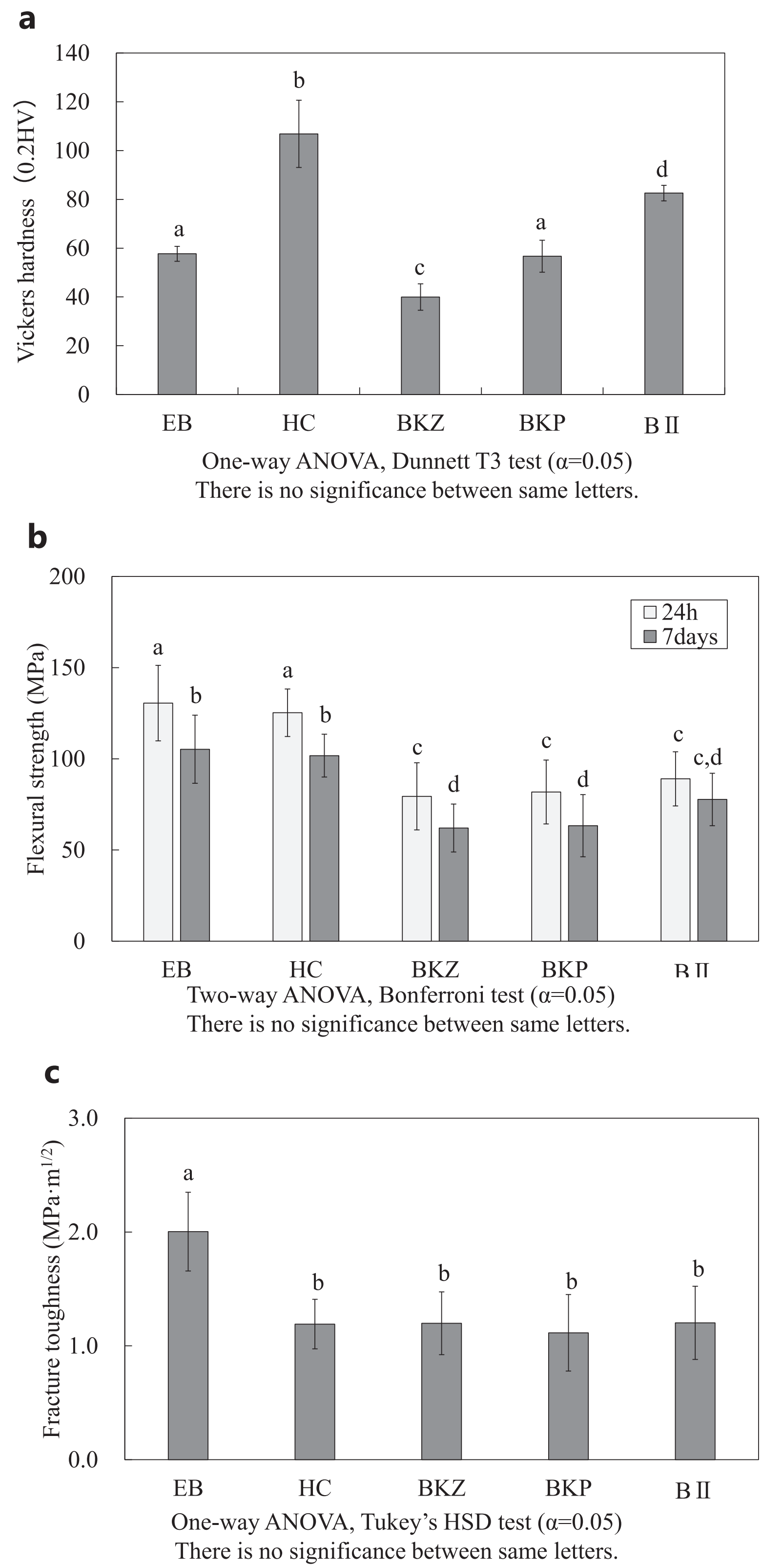

Fig. 1 – a. Vickers hardness of computer-aided design/computer aided manufacturing (CAD/CAM) resin composites (EB and HC) and other resin composites. b. Flexural strength of CAD/CAM resin composites (EB and HC) and other resin composites after 24 h/7 days of water immersion. c. Fracture toughness of CAD/CAM resin composites (EB and HC) and other resin composites.

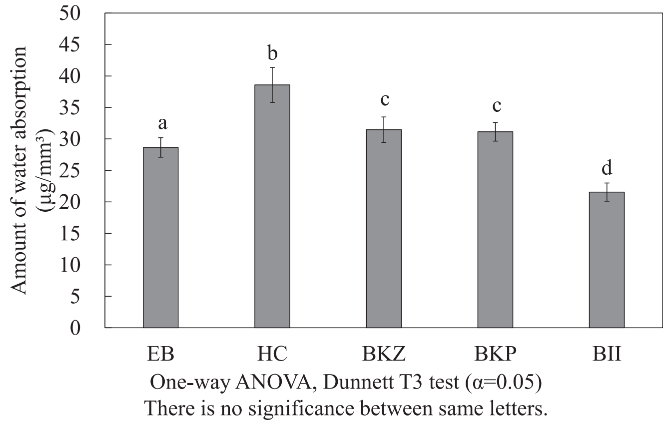

Fig. 2 – Water absorption of computer-aided design/computer aided manufacturing (CAD/CAM) resin composites (EB and HC) and other resin composites.

between hardness and the elastic modulus [19], and the elastic modulus is also related to the stiffness of a material [20]. These reports suggest that EB, with a low hardness score, will exhibit a low elastic modulus and high fracture toughness. The fracture toughness of EB was higher than that of the other materials. CAD/CAM RCBs have greater mechanical durability than resin composites for permanent teeth [21], and EB, with a higher fracture toughness than HC, is useful for the restoration of thin primary molar teeth. The fracture toughness of EB was 2.0 ± 0.35 MPa·$m^{1/2}$, which is higher than that of commercially available CAD/CAM RCBs developed for premolar restorations (0.60 ± 0.12 MPa·$m^{1/2}$ to 1.0 ± 0.32 MPa $m^{1/2}$) [22]. Premolar teeth are the successor teeth of primary molar teeth, suggesting that the fracture toughness of EB is acceptable for the restoration of primary teeth.

While the flexural strength of all materials except BII decreased following water immersion, the flexural strength of EB satisfied the requirement for resin composite (Type 1, Class 2, Group 2: more than 100 MPa after water immersion for 7 days) as defined in ISO 4049:2019. The water absorption of EB satisfied the requirement for resin composite (less than 40 μg/mm) as defined in ISO 4049:2019. Because BII recorded the lowest water absorption, there was little decrease in the flexural strength of BII. The development of an alternative monomer with decreased water degradation [23] would potentially improve the flexural properties of EB after water immersion.

Differences in the composition of restorative materials affect antagonist enamel wear [24], and the wear rate of a restorative material should be similar to that of enamel [25]. Previous studies on primary tooth abrasiveness against restorative materials have focused on resin composites [26–28]. Primary tooth wear commonly occurs when the enamel is lost and dentin is exposed on the occlusal surface [29,30]. The abrasive performance of primary and permanent teeth depends on the strength of the enamel [31], morphological factors such as enamel and dentin thickness [32], and differences in the biting force of adults and children [33]. The physical properties of enamel, parafunctional habits, eating habits, and antagonist materials have been reported to affect clinical wear [34–37]. A positive correlation in terms of wear resistance has been reported between the surface hardness of materials and the depth of wear of the antagonist enamel [38]. The antagonist wear of EB was lower than that of HC, and SEM analysis revealed the smooth surface of EB. Greater antagonist wear is induced by large filler particles in resin composites [39], suggesting that the smaller filler particles of EB resulted in improved compressive strength and a smooth surface in the antagonist enamel [40]. The occlusal preparation for CAD/CAM resin composite crowns for permanent teeth is recommended to be greater than 1.5 mm. Taking this recommendation into account, the acceptable occlusal preparation for crowns on primary teeth should be less than 1.0 mm (1000 μm) because primary teeth are thinner than permanent teeth. The amount of wear for EB (43.9 ± 9.88 μm)

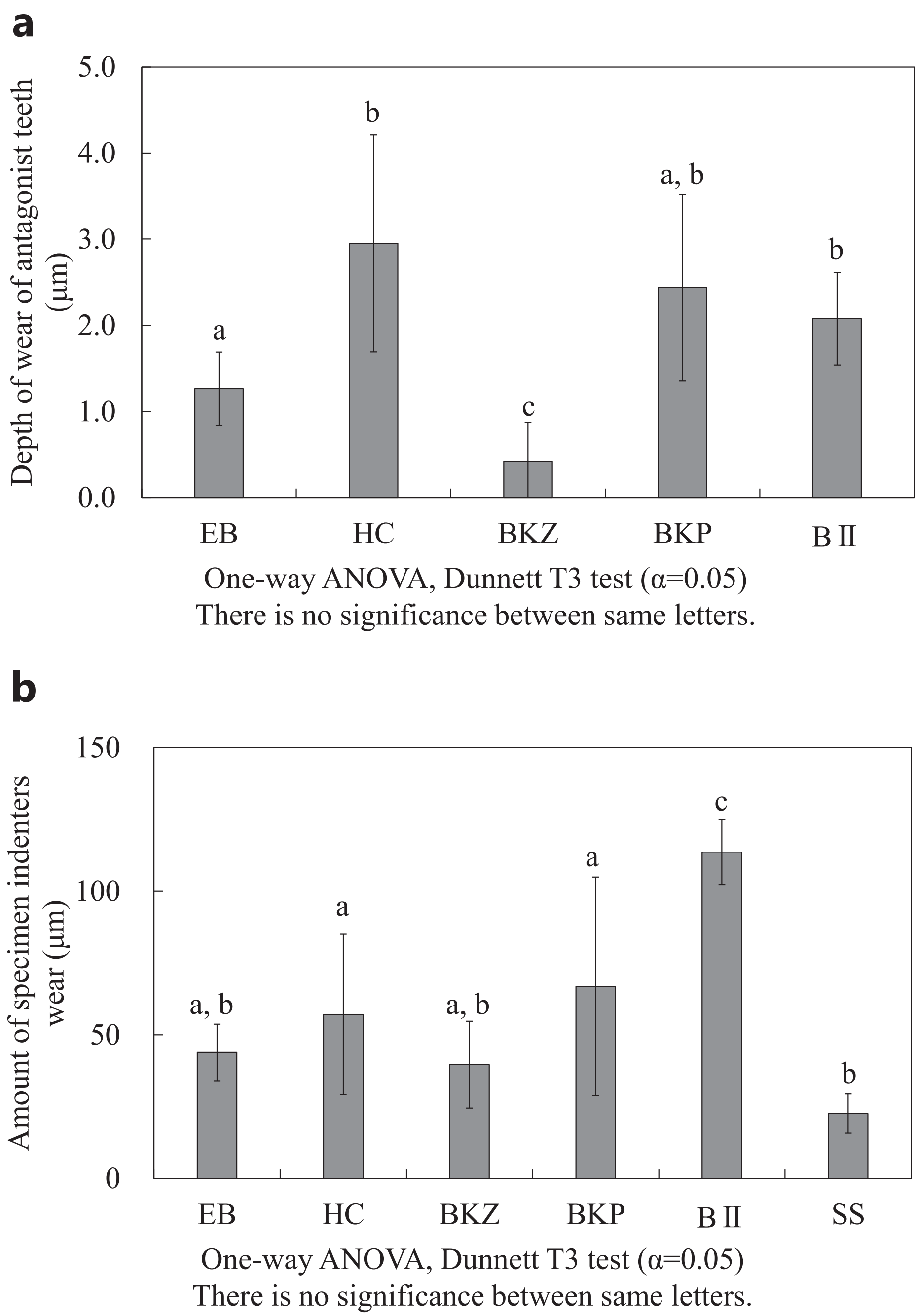

Fig. 3 – a.Depth of wear of antagonist teeth for computer-aided design/computer aided manufacturing (CAD/CAM) resin composites (EB and HC) and other resin composites. b. Amount of wear of CAD/CAM resin composites (EB and HC), other resin composites, and stainless steel.

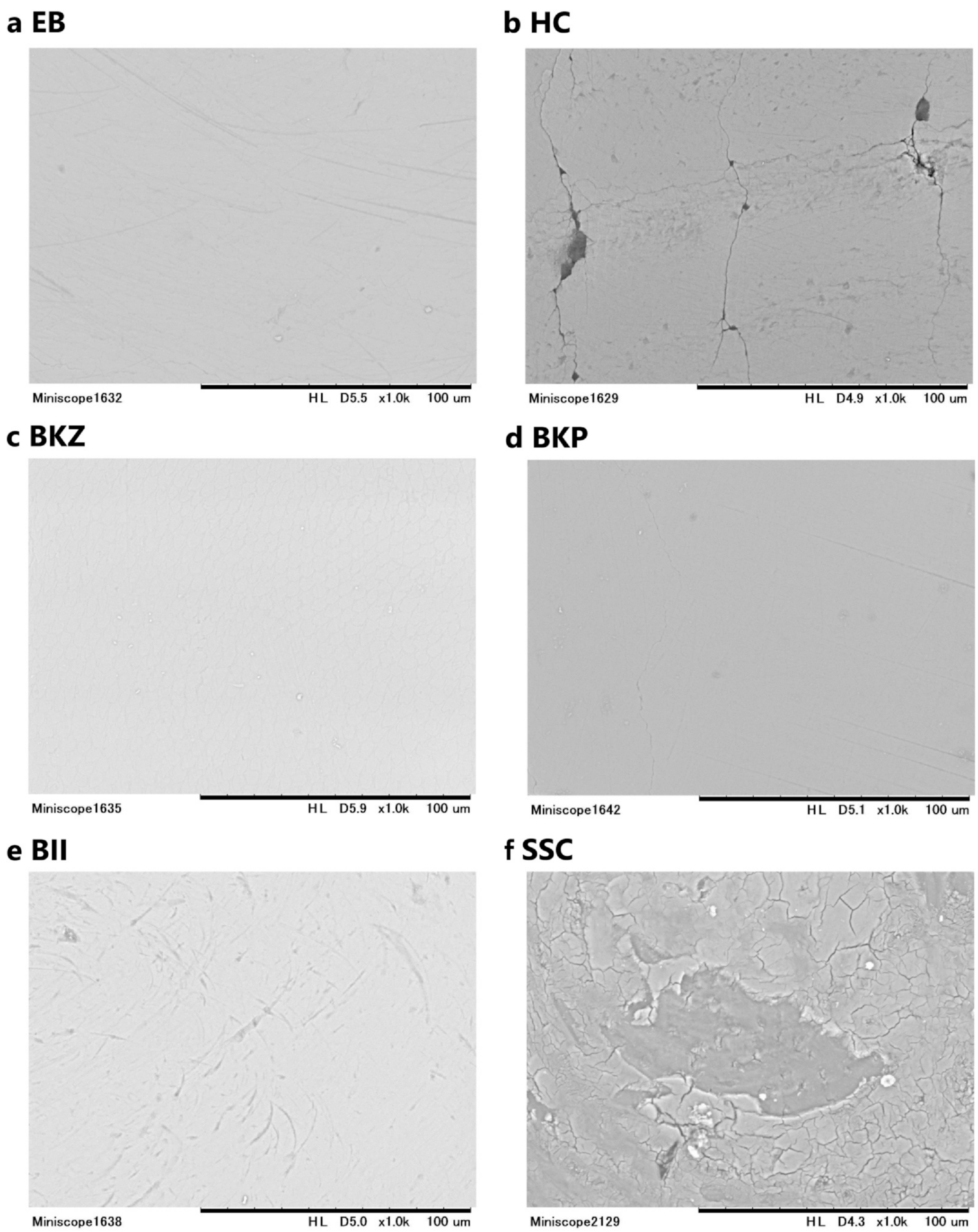

Fig. 4 – (a) Scanning electron microscope images of EB, (b) HC, (c), BKZ, (d) BKP, (e) BII, and (f) SSC.

was certainly less than the amount of preparation, indicating that EB has sufficient wear resistance.

Within the limitations of this *in vitro* study, acceptable physical properties and wear resistance against primary molar teeth were confirmed. Primary teeth have been found to be less mineralized than permanent teeth [41]. The S-PRG filler used in this study may protect primary teeth from demineralization. A further investigation is ongoing evaluating the ion release profile and bio-functional abilities (such as antibacterial activity) of CAD/CAM RCBs containing S-PRG filler. CAD models for non-linear dynamic finite element analysis of CAD/CAM resin composite crowns for primary teeth is under development to define the initial load for fatigue tests to clarify the longevity of CAD/CAM RCBs [42,43].

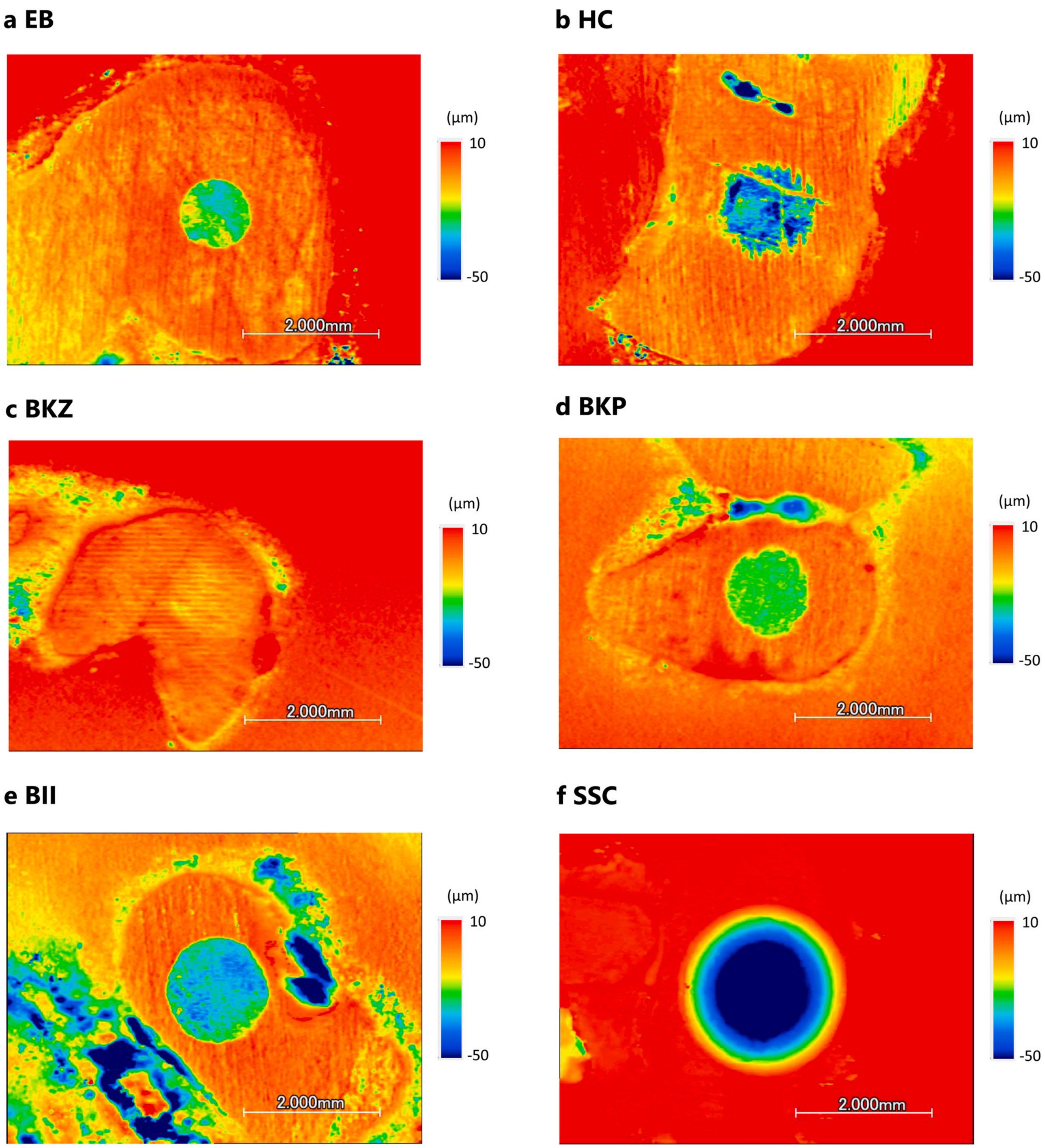

Fig. 5 – Three-dimensional microscope images of antagonist enamel for (a) EB, (b) HC, (c) BKZ, (d) BKP, (e) BII, and (f) SSC.

5. Conclusion

The CAD/CAM RCB containing S-PRG filler we developed for primary molar teeth demonstrated adequate physical properties and wear performance, suggesting that this material is suitable for restoration of primary molar teeth and can be used as an alternative to stainless steel crowns.

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