Acknowledgements

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The effect of dentine surface preparation and reduced application time of adhesive on bonding strength

Objective. This study evaluated the effects of surface preparation and the application time of adhesives on the resin-dentine bond strengths with universal adhesives.

Methods. Sixty molars were cut to exposed mid-coronal dentine and divided into 12 groups (N=5) based on three factors; (1) adhesive: G-Premio Bond (GP, GC Corp., Tokyo, Japan), Clearfil Universal Bond (CU, Kuraray Noritake Dental Inc., Okayama, Japan) and Scotchbond Universal Adhesive (SB, 3M ESPE, St. Paul, MN, USA); (2) smear layer preparation: SiC paper ground dentine or bur-cut dentine; (3) application time: shortened time or as manufacturer's instruction. Fifteen resin-dentine sticks per group were processed for microtensile bond strength test (µTBS) according to non-trimming technique (1 mm^2) after storage in distilled water (37ºC) for 24 h. Data were analyzed by three-way ANOVA and Dunnett T3 tests (α=0.05). Fractured surfaces were observed under scanning electron microscope (SEM). Another 12 teeth were prepared and cut into slices for SEM examination of bonded interfaces.

Results. µTBS were higher when bonded to SiC-ground dentine according to manufacturer's instruction. Bonding to bur-cut dentine resulted in significantly lower µTBS ($p<0.000$). Shortening the application time resulted in significantly lower bond strength for CU on SiC and GP on bur-cut dentine. SEM of fractured surfaces revealed areas with a large amount of porosities at the adhesive resin interface. This was more pronounced when adhesives were bonded with a reduced application time

and on bur cut dentine.

Clinical significance: The performance of universal adhesives can be compromised on bur cut dentine and when applied with a reduced application time.

Key words: Universal adhesives, Microtensile bond strength, Bonding application time, Surface preparation, SEM

1. Introduction

All-in-one self-etching adhesives have become popular in dentistry because of their advantages such as less technique sensitivity and user-friendliness[1]. However, there are still concerns about the effectiveness of 1-step adhesives when bonding to uncut enamel[2, 3], to different smear layer preparation[4, 5] and their long-term durability[1].

Recently, universal adhesives have been introduced to the market. They are principle 1-step self-etching adhesives that can be applied in either self-etching mode or etch-and-rinse mode[6-8]. Similar to the mildly acidic self-etching adhesives, there are concerns regarding the effect of smear layer on their bonding performance[5, 9, 10]. Most of the studies that have evaluated the effect of smear layer preparation on the bond strength of adhesives used only different SiC-paper grits, which can be regarded as not clinically relevant[9-16]. Furthermore, the study from Oliveira *et al*[9] reported that the loosely organized smear layer produced by SiC papers was easier for self-etching primer to penetrate when compared to those of diamond burs.

To overcome the infiltration-impairing effect of smear layer, prolonged application time has been suggested as an option to increase bond strength[11]. Although, the bond strength improvement might be system-specific[12]. However, contrary to the suggestive findings referred above, the newly developed product from GC Corp., G-Premio Bond claims that high bond strength can be achieved even when applied with shortened application time (optional manufacturer's instructions). Although shorter application time may be clinically appealing, the procedure may carry negative consequences to adhesive infiltration and solvent evaporation. Since one product has adopted such optional reduced application time, it becomes interesting to test if such approach can also be applied to other adhesives; and whether the type of smear layer plays a role on the adhesive effectiveness at different application times.

Therefore, the aim of this study was to evaluate the effects of adhesive application time and dentine surface preparation on resin-dentine microtensile bond strength (µTBS) of three universal adhesives. The null hypotheses tested were that 1) there is no effect of application time and, 2) there is no effect of surface preparation on the bond strength.

2. Materials and methods

2.1 Tooth selection and preparation

Seventy-two extracted non-carious human third molars were used in this study. They were stored in an aqueous solution of 0.5% Chloramine-T at 4ºC and used within 6 months after extraction. The teeth were collected under a protocol reviewed and approved by the university ethical committee (#2013-7). The teeth were abraded to expose mid-coronal dentine with a gypsum model trimmer under water coolant. A light microscope was used to confirm that no enamel remained on the dentine surface.

2.2 Adhesives and bonding procedures

The teeth were randomly assigned into 12 experimental conditions (n=5 to µTBS;

n=1 to interfacial structure observation) according to: dentine surface preparation (SiC-prepared dentine vs. bur-cut dentine) and adhesive application time (manufacturer's instruction vs. shortened). These variables were tested for three adhesive systems: G-Premio Bond [GP, GC Corp., Tokyo, Japan], Clearfil Universal Bond [CU, Kuraray Noritake Dental Inc., Okayama, Japan], and Scotchbond Universal Adhesive [SB, 3M ESPE, St. Paul, MN, USA]. Details of the variables and products can be found in Table 1.

Occlusal dentine surfaces were prepared by using either 600-grit SiC paper (Sankyo-Rikagaku Co., Saitama, Japan) or tapered regular grit diamond bur (diamond point FG, #103R, Shofu, Kyoto, Japan). For SiC paper preparation, the surfaces were manually polished for 60s under running water using a 600-grit SiC paper. In case of diamond bur, dentine surfaces were ground with the bur in a high-speed handpiece with copious water spray for 5 light-pressure strokes per tooth in order to make a uniform surface. For each surface preparation, half of the teeth received the adhesives applied according to manufacturer’s instruction, and the other half received the adhesives applied under the shortened time. Each adhesive was dropped directly from the bottle on dentine, air dried immediately and then light cured. Two 2mm-thick layers of resin composite (Clearfil AP-X, Kuraray Noritake Dental Inc., Tokyo, Japan) were built-up on the bonded surface. Each layer was light cured for 20 s operating using a light curing device (Optilux 401, Demetron/Kerr, Orange, CA, USA) at ≥550 mW/cm^2.

2.3 Microtensile bond strength (µTBS) test

After storage in 37°C water for 24 h, each bonded tooth was sectioned into beams (cross-sectional area approximately 1mm^2) using an Isomet diamond saw (Isomet 1000, Buehler, Lake Bluff, Illinois, USA). For each tooth (n = 5), three beams from the central area were randomly selected for µTBS, therefore resulting in a total of 15 beams to be tested. The remainder of the beams was stored for longer-term testing. The beams were fixed to a Ciucchi's jig with cyanoacrylate glue (Model Repair 2 Blue, Dentsply-Sankin, Otahara, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/min in a desktop testing apparatus (EZ test, Shimadzu, Kyoto, Japan). µTBS was expressed in MPa, and data were analyzed by three-way ANOVA and Dunnett T3 tests (α=0.05).

2.4 Fracture mode analysis

The fractured specimens were mounted on an aluminum stub, then coated with Pt-Pd for 150 seconds. The fracture modes were determined using a scanning electron microscope (SEM, S-4000, Hitachi, Tokyo, Japan) at an accelerating voltage of 10 kV. Surfaces were examined at lower magnification to categorize the mode of fractured and specific features were further examined at 3000× and 10000×. Fracture mode categories were classified into four groups [13]: A, adhesive failure; CD, cohesive failure within dentine; CC, cohesive failure within composite resin; or M, mixed failure.

2.5 Interfacial structure observation

One tooth per group was bonded in the same way as described for μTBS test. The teeth were cut parallel to the long axis into slabs. Two slabs from central part were selected and prepared for SEM observation by following a protocol described by Ting *et al* [14]. All slabs were serially polished with the series of SiC papers and diamond pastes. After that, treated with 5% HCl for 30s followed by NaOCl for 5 min. Then, the slabs were left to air dry for 24h. Finally, they were sputter-coated with Pt-Pd for 150 seconds and then examined at 3000× magnification.

3. Results

3.1 μTBS

There were no pre-test failures in this study. Our results indicated that there were significant effects between adhesive vs surface preparation ($F=12.02$; $p<0.000$), and adhesive vs application time ($F=3.5$; $p=0.032$). There was no direct effect of surface preparation vs application time ($F=1.17$; $p=0.280$). The interaction of factors was significant ($F=10.006$; $p<0.000$)

Bond strengths were always significantly higher when the adhesives were bonded to 600-grit SiC paper-prepared surfaces than when bonded to bur-cut dentine, regardless of the application time (Table 2). The influence of application time was only observed for CU and GP and was dependent on the surface preparation. CU presented significantly lower bond strengths when bonded using a shortened application time on SiC paper prepared dentine; and GP resulted in significantly lower bond strengths when used with the short application mode on the bur-cut dentine (Table 2).

3.2 Fracture modes

In general, the fracture modes were mainly categorized as mixed failure and adhesive failure. There was a clear tendency that more cohesive failures occurred with SiC prepared dentine (Fig 1). The SEM examination of the fracture modes revealed unusual features on the fractured surfaces. When adhesive failure areas were examined at higher magnifications (10000× and 3000× (inserts)), a high concentration of porosity was observed for both CU and GP adhesives, and at a lower degree for SB adhesive. More porosity and bigger pores appeared to be associated with the groups that were bonded with shortened application time and on bur-cut dentine (Fig. 2, additional images of SiC prepared dentine are available online). The pores were predominantly round having a submicron sized diameter. Only those in GP were above 1-2 microns. They were uniformly distributed on the entire surface of the adhesive failure beams with some areas presenting a concentration of larger pores.

3.3 Interfacial structure observation

The representative of SEM images were showed in Fig. 3-5. A general observation for all groups was the fact that the hybrid layer was not distinct from the SEM images (Fig. 3-5). Resin tags detected were short, sparsely distributed and only more distinct on surfaces prepared with SiC paper and preferably when the adhesive was applied according to the manufacturer's directions (Fig. 3b, 4b, 5b). Resin tags were either absent or appeared as very short projections and scarcely distributed along the observed area when the adhesives were bonded to bur-cut dentine, regardless of the application time (Fig. 3c,d, 4c,d, 5c,d). Round voids were sometimes encountered within the adhesive layer of GP applied to bur-cut dentine and under the shortened time (Fig. 4c). In some areas, GP adhesive layer appeared to have two distinct layers (visible by different contrast in the images) separated by a jagged line with some round shape features (Fig. 4a).

4. Discussion

According to the results, the type of surface preparation and the adhesive application time both had a significant effect on the bond strengths. Therefore, both of the anticipated null hypotheses were rejected. Many laboratory studies evaluate adhesive systems with bond strength testing. Most of them prepared dentine surface by using SiC paper to reproduce the smear layer in clinical situation[15-17]. However, it is expected that the characteristics of the smear layer will vary according to the preparation variables[9, 10]. Moreover, it is expected that the resultant smear layer will have an impact on the performance of the adhesive system, particularly on the so-called mild self-etch category[1, 18]. It has been demonstrated that smear layers prepared with a diamond bur were more compact than those prepared by SiC paper when examined under SEM[9, 10] and TEM[5]. Therefore, in the case of self-etch adhesives, it is possible that the denser smear layer might hinder the acidic monomer infiltration, hence compromising the bond strength. The results of present study also supported this hypothesis. Bonding to bur-cut dentine always resulted in significantly lower µTBS, regardless of the adhesive or application time. According to the interfacial analysis, it appeared that resin tags were more evident and apparently better formed when the adhesives were bonded to surfaces prepared by SiC paper following the manufacturer's recommended application time (compare Figs 3b vs 3a,c,d; 4b vs 4a,c,d; 5b vs 5a,c,d).

Without phosphoric acid etching, acidic monomers of self-etch adhesives do not remove the smear layer. Rather, the smear layer is partially demineralized and incorporated into the hybrid layer. Recently, Mine *et al*[5] demonstrated that there are two zones of hybridized layers when ultra-mild one-step self-etch adhesives are used. The upper portion is called “resin smear complex”, which is the result of resin infiltration into the residual surface smear. The lower, thinner portion is the “true hybrid layer". From their observation, the adhesive resin was able to penetrate the SiC ground smear layer more effectively than the bur-cut smear layer. When the acidic monomers from self-etch adhesives attempt to infiltrate across the smear layer, they are simultaneously buffered by the minerals present within the smear layer and, therefore, gradually loose their acidity and capacity to self-infiltrate. In thicker, more compact bur-cut smear layer, the acidic monomer of the adhesives might had not been able to penetrate uniformly across the smear layer to form the true hybrid layer with the underlying dentine. This possibility supports the lower bond strengths and the fracture modes that were mainly mixed and adhesive failure on bur-cut dentine. In our study, the type of smear layer had the most significant impact on the bond strength.

Significant reductions in bond strengths were always observed for bur-cut dentine, regardless of the adhesive type used or mode of application. This finding has profound clinical implications since bur-cut dentine is usually the type of dentine clinicians encounter in daily practice. Therefore, significantly lower bond strengths should be expected when bonding clinically with the adhesives evaluated in this study.

In this study, multiple functional monomer containing adhesives were tested (4-MET, 10-MDP, and MDTP in GP; polyalkynoic acid copolymer and 10-MDP in SB). These multiple functional monomers might improve the bonding performance by interacting to the compact bur-cut smear layer. This could explain the higher µTBS of GP and SB on bur-cut dentine.

Some studies have recommended a prolonged application time to increase the bond strengths of self-etch adhesives[11, 12, 19]. As the application time is extended, the increase in monomer infiltration might be expected[19, 20]. However, prolonged application times might not be practical in clinics. On the contrary, clinicians in general would desire to reduce the application time. In this study, in order to understand the effect of the application time on the bond strength, the application time was experimentally minimized by blowing the adhesive immediately after directly dropping the adhesive from the bottle on the dentine surface. The overall results demonstrated that adhesive application according to manufacturer's instruction (longer) provided higher µTBS. However, statistically significant differences were observed only in case of CU on SiC and GP on bur-cut dentine. For SB, reduction in application time has no influence on μTBS regardless of prepared surface. However, pores on fractured surface was less pronounced when SB was applied according to manufacturers' instruction (Fig 2f). Furthermore, resin tags were better formed compared to shortened application time (Fig 5a-d). These might be caused by the longer application time (20s) associated with rubbing motion according to the manufacturers' instruction (Table 1). This is also reported by Amsler *et al* [21] that reduced application time had no effect on bonding performance of SB.

As discussed above, the infiltration of the self-etch adhesive across the smear layer is a time dependent process, which is also hindered by the buffering action of the smear layer[4]. It is expected, therefore, that the shorter application time might have not been sufficient for the acidic monomer to infiltrate across the smear layer and form a strong bond with underlying dentine. Residual solvent and water entrapped within the adhesive layer may have also played a role on the results. With shortened application time, solvent evaporation might have not been sufficient. Therefore, the residual solvent could have compromised the adhesive polymerization[22, 23] and, therefore, the resultant bond strength. Erhardt *et al*[12] stated that the effect of prolonged application time was system dependent. Extended application time cannot improve solvent evaporation when water is added as a solvent into ethanol solvated monomer. Both water and ethanol can hydrogen bond to each other and also to monomers. Thus, their evaporation rate is reduced[24]. In current study, the examination of adhesive failures that occurred at the interface between the adhesive and the resin revealed a large concentration of submicron-size pores (Fig. 2).

These features were highly evident in the groups that were applied with a shortened time (Fig. 2a,c,e). We believe these pores represent entrapped solvent and water that could not evaporate due to the limited amount time allowed. Similar features have been reported on interfaces examined by TEM. The authors suggested that such round pores are a result of droplets caused by phase separation of the adhesive that rendered entrapped within the adhesive layer. Furthermore, they suggested that the shorter application time was not enough for droplet evaporation[25].

Although the presence of this large amount of voids at the interface between the resin and the adhesive has reduced the bonded surface are dramatically, it is surprising that the bond strengths did not fall significantly, except for 2 groups (Table 2). The presence of the voids, however, appears to have driven the failures to occur at that interface. While no remarkable compromising effect was observed because of the presence of the voids, however, a crucial effect on the long-term adhesion could be expected.

The results of this study have important clinical implications. To purposely reduce the application time of the adhesives used in this study is not a recommended practice. Quick application time increase the risk of entrapment of solvents within the adhesive that might have profound consequences for the degradation of the bonded assembly after water sorption. The risk of compromised bonding with reduced application time increases when dentine is prepared by bur, which is almost always the case in a clinical setting.

5. Conclusions

Within the limitation of this study, it may be concluded that

1. Dentine surface preparation had an influence on the μTBS. Smear layer form bur-cut dentine had an undesirable effect on all the three universal adhesives used in this study.
2. Application time had an impact on the adhesive performance. The shortened application time resulted in insufficient solvent evaporation and bonding mechanism which leads to lower bond strength for two out of three adhesives tested, depending on the type of surface preparation.

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The effect of dentine surface preparation and reduced application time of adhesive on bonding strength

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Key words: Universal adhesives, Microtensile bond strength, Bonding application time, Surface preparation, SEM

Table 1 Adhesive system (batch number), composition and application procedures.

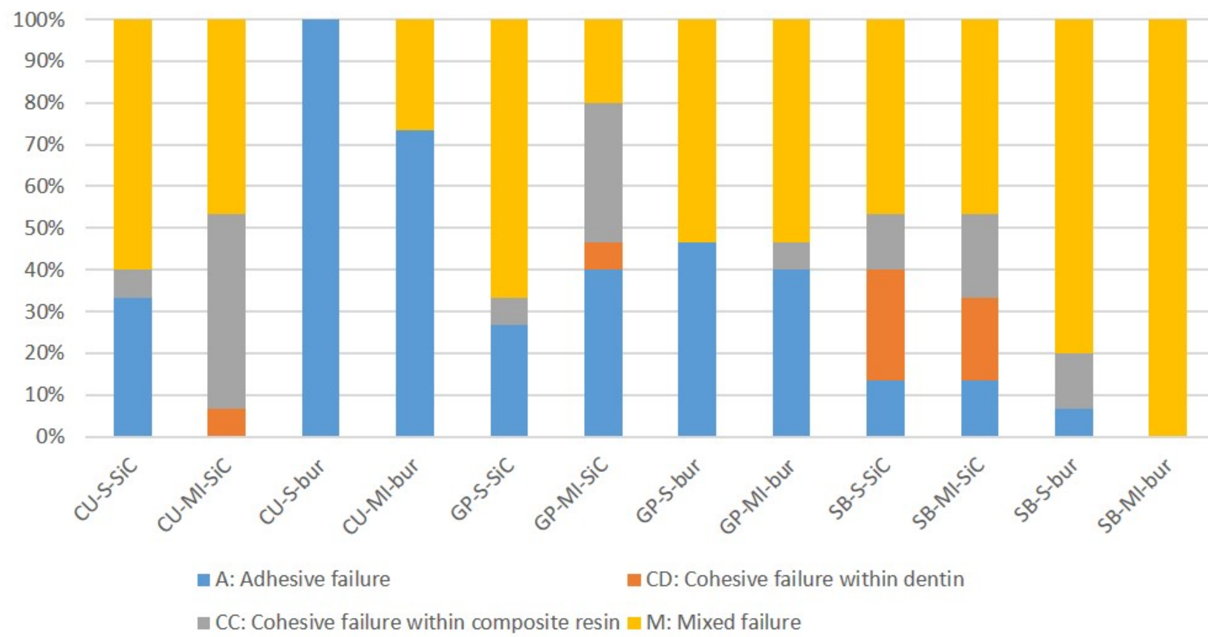
Adhesive (batch number)	pH*	Composition	Manufacturers' instruction
Clearfil Universal Bond (000002)	2.3	10-MDP, Bis-GMA, HEMA, ethanol, hydrophilic aliphatic dimethacrylate, colloidal silica, dl-camphorquinone, silane coupling agent and water.	1. Apply the adhesive to the dentin surface with the applicator brush and rub it in for 10 s. 2. Dry the dentin surface sufficiently by blowing mild air for more than 5 s until the adhesive does not move. 3. Light cure for 10 s.
G-Premio Bond ** (1411061G)	1.5	10-MDP, 4-META, 10-methacryoyloxydecyl dihydrogen thiophosphate, methacrylate adic ester, distilled water, acetone, photo initiators, silica fine powder.	1. Apply using a microbrush 2. Leave undisturbed for 10 s after application. 3. Dry thoroughly for 5 s with oil free air under maximum air pressure. 4. Light cure for 10 s.
Scotchbond Universal (572054)	2.7	10-MDP, HEMA, silane, dimethacrylate resins, Vitrebond™ copolymer, filler, ethanol, water, initiators.	1. Apply the adhesive on the surface and rub it in for 20 s. 2. Gently air-dry the adhesive for approximately 5 s for the solvent to evaporate. 3. Light cure for 10 s.

10-MDP: 10-methacryloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A diglycidyl methacrylate;
HEMA: 2-hydroxyethyl methacrylate; 4-META: 4-methacryloyloxyethyl trimellitate anhydrate
* The pH for SB and CU was obtained from ref [6]. For GP it was informed by the manufacturer.
** The shortened application time is an optional application mode suggested by the manufacturer.

Table 2 Bond strength of adhesives according to surface preparation and application time. Values are expressed in MPa (SD).

Adhesive	SiC		bur	
	Shortened application time (S)	Manufacturer's instruction (MI)	Shortened application time (S)	Manufacturer's instruction (MI)
Clearfil Universal Bond	$48.6(11.8)^{b,c,d}$	$66.3(10.4)^{a}$	$14.7(4.8)^{f}$	$19.6(6.3)^{f}$
G-Premio Bond	$61.6(7.9)^{a,b}$	$63.3(12.1)^{a}$	$28.7(6.7)^{e}$	$46.4(7.9)^{c}$
Scotchbond Universal Adhesive	$68.6(11.1)^{a}$	$68.9(10.6)^{a}$	$29.1(6.7)^{e}$	$35.1(7.4)^{d,e}$

Different superscript letters indicate statistically significant differences ($p<0.05$).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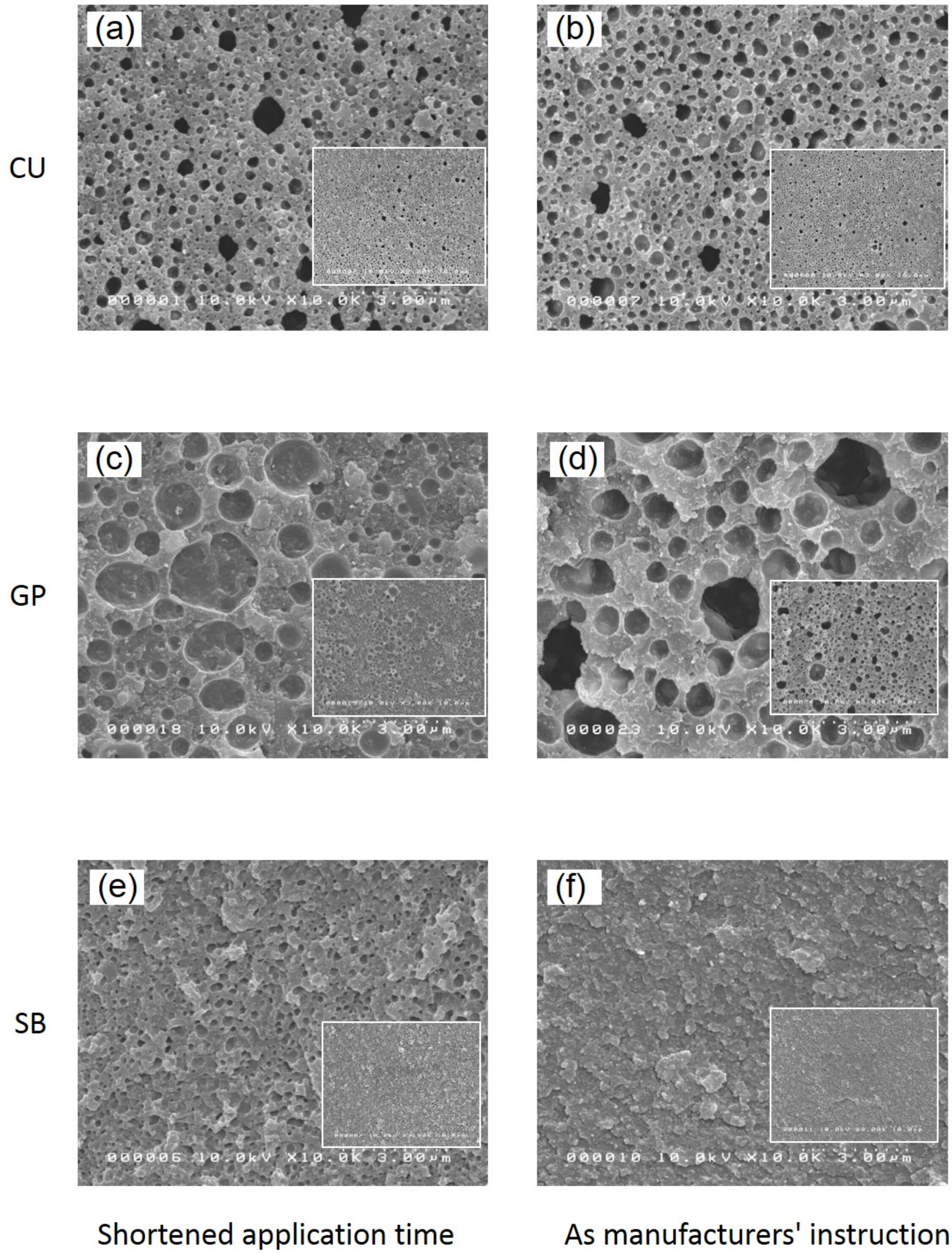
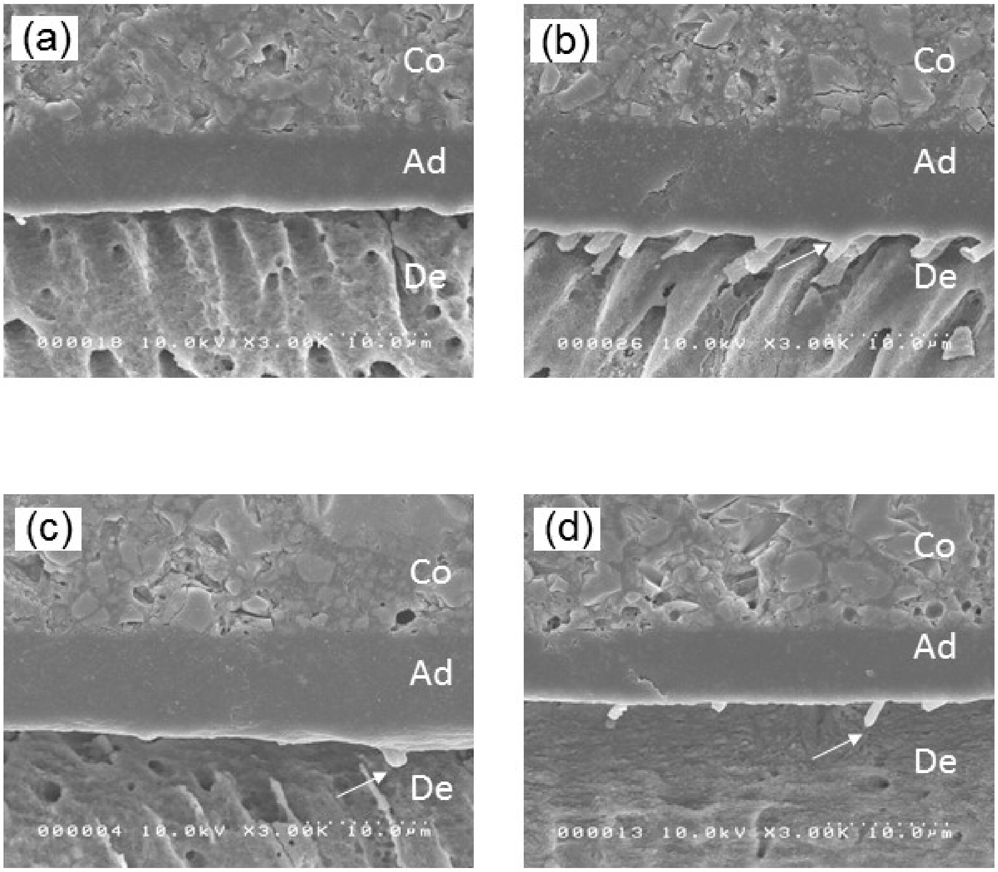
CU-MI-SiC
CU-S-bur
CU-MI-bur
GP-S-SiC
GP-MI-SiC
GP-S-bur
GP-MI-bur
SB-S-SiC
SB-MI-SiC
SB-S-bur
SB-MI-bur
A: Adhesive failure
CD: Cohesive failure within dentin
CC: Cohesive failure within composite resin
M: Mixed failure



(a)
(b)
(c)
(d)
(e)
(f)
CU
GP
SB
000001 10.0kV X10.0K 3.00μm
000007 10.0kV X10.0K 3.00μm
000018 10.0kV X10.0K 3.00μm
000023 10.0kV X10.0K 3.00μm
000006 10.0kV X10.0K 3.00μm
000010 10.0kV X10.0K 3.00μm
Shortened application time
As manufacturers' instruction

SiC

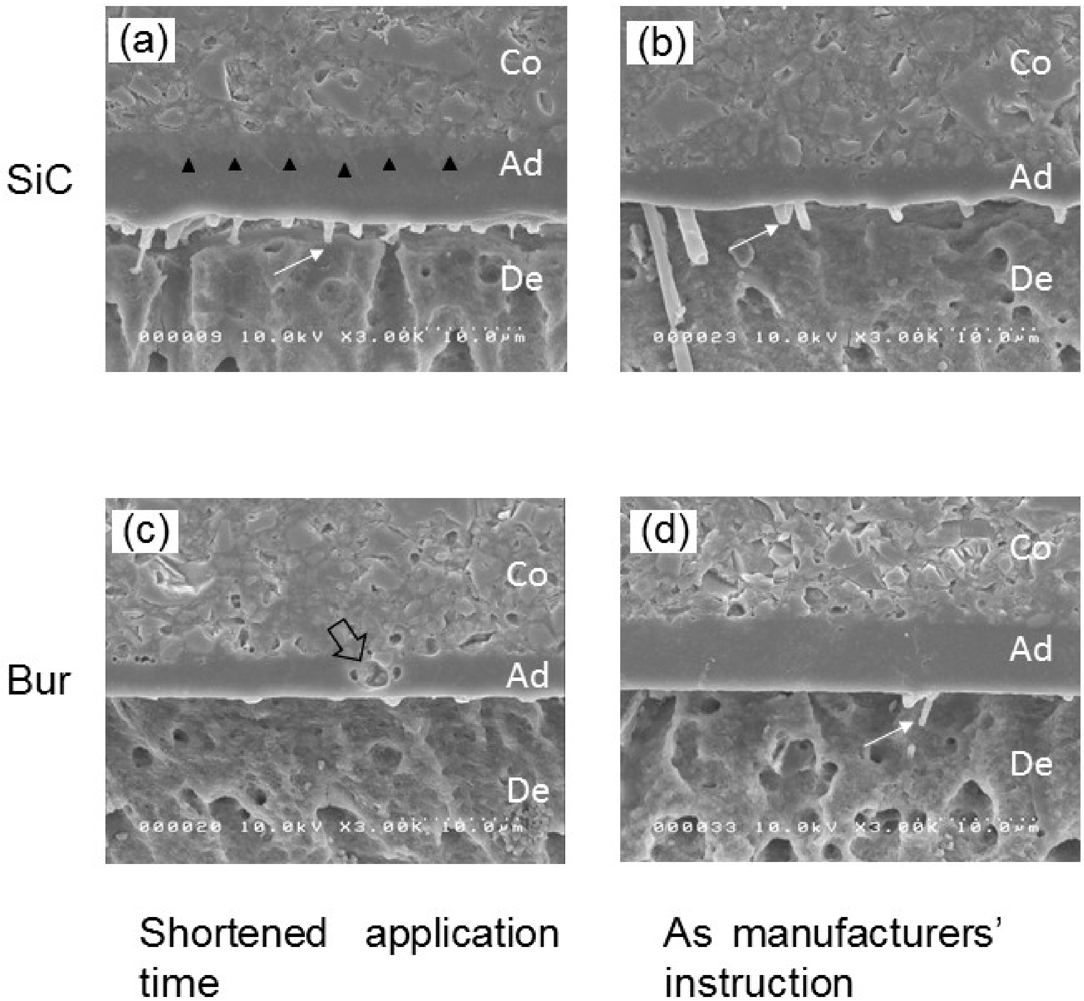
Bur



Shortened application time

As manufacturers' instruction

CU



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(a)
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000020 10.0kV X3.00K 10.0µm
(d)
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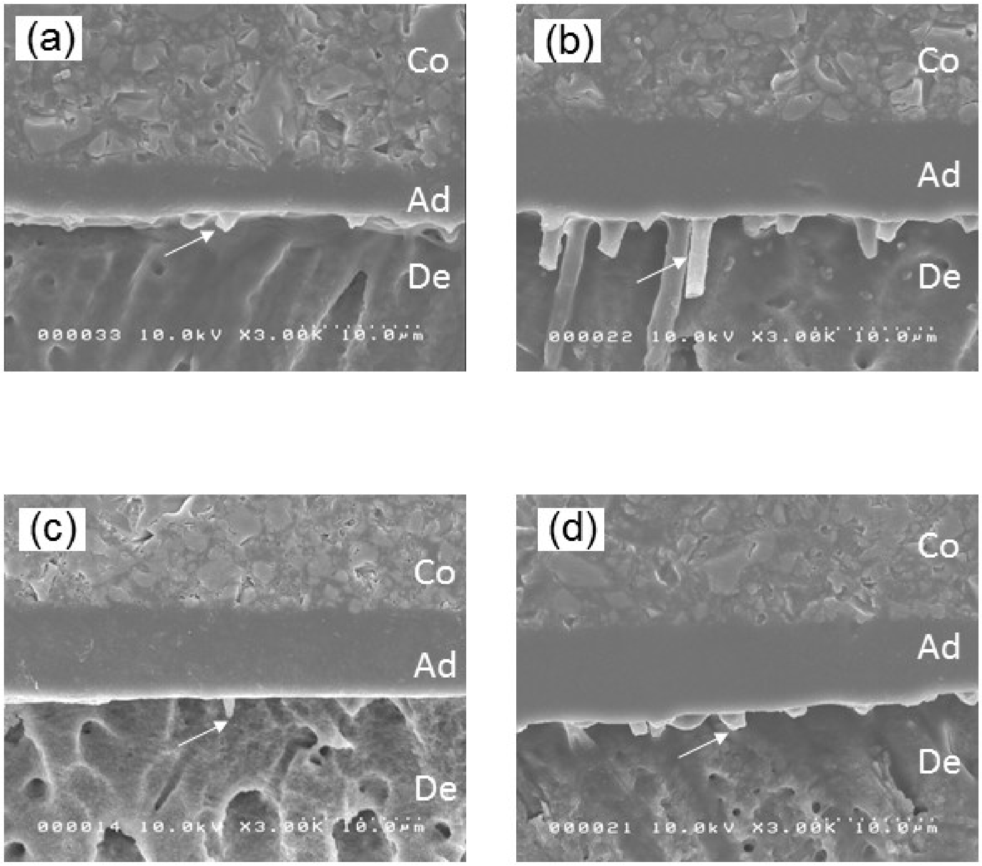
000033 10.0kV X3.00K 10.0µm
Shortened application time
As manufacturers' instruction

GP



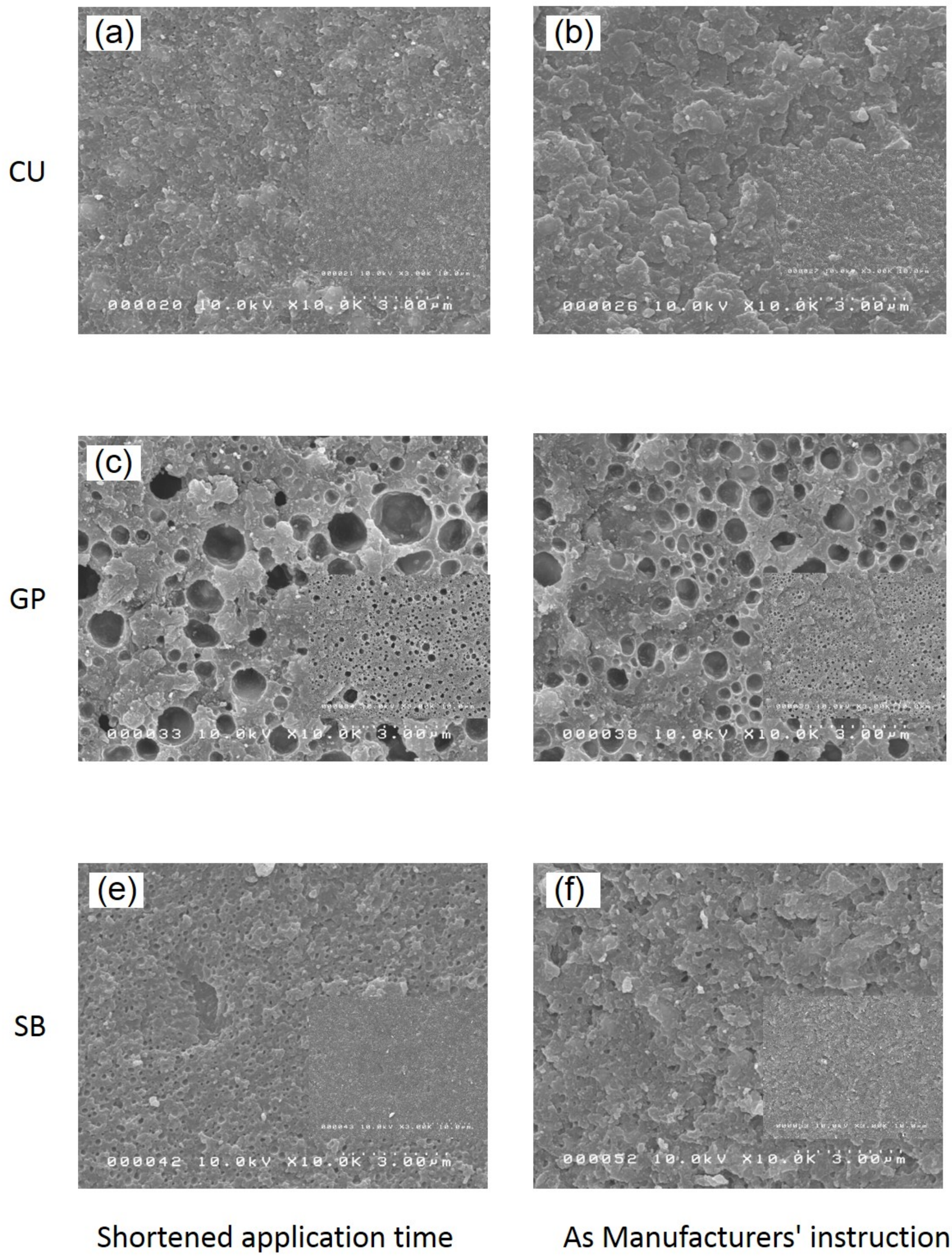
SiC

Bur

Shortened application time

As manufacturers' instruction

SB


(a)
000020 10.0kV X10.0K 3.00μm
(b)
000026 10.0kV X10.0K 3.00μm
CU
(c)
000033 10.0kV X10.0K 3.00μm
000038 10.0kV X10.0K 3.00μm
GP
(e)
000042 10.0kV X10.0K 3.00μm
(f)
000052 10.0kV X10.0K 3.00μm
SB
Shortened application time
As Manufacturers' instruction