

## Laboratory Research

# Effect of Different Bur Grinding on the Bond Strength of Self-etching Adhesives

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### Clinical Relevance

In some self-etching systems, selecting the proper bur type for cutting dentin is important for improving bond strength.

### SUMMARY

**This study compared the microtensile bond strength (MTBS) of three all-in-one adhesive systems and a two-step system using two types of burs to prepare the dentin surfaces. Flat coronal**

**surfaces of 24 extracted human molars were produced using either regular-grit or superfine-grit diamond burs. Resin composite was then bonded to equal numbers of these surfaces using one of the four adhesives: Clearfil SE Bond (CSE), G-Bond (GB), SSB-200 (SSB) or Prompt L-Pop (PLP). After storage for 24 hours in 37°C distilled water, the bonded teeth were sectioned into slices (0.7-mm thick) perpendicular to the bonded surface. The specimens were then subjected to microtensile testing and the bond strengths were calculated at failure. Bond strength data were analyzed by two-way ANOVA and the Games-Howell test for interaction between adhesive and type of cut dentin. The fractured surfaces were observed by SEM to determine the failure mode. In addition, to observe the effect of conditioning, equal numbers of the two bur-cut dentin surfaces of eight additional teeth were conditioned with the adhesives and observed by SEM. Based on the results, when CSE and SSB were bonded to dentin cut with a regular-grit diamond bur, the MTBS values were significantly lower than that of superfine bur-cut dentin; whereas, GB and PLP showed no significant differences in MTBS between the two differently cut surfaces. SEM observation of the fractured surfaces revealed a mixed mode (adhesive in some areas and cohesive in others in the same sample) of failure in all specimens except PLP, which showed cohesive failure within the adhesive for both types of bur**

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**preparation. Generally, SEMs of the conditioned surfaces using both types of burs showed partial removal of the smear layer for CSE, minimal for GB and SSB and complete removal for PLP.**

**In conclusion, when cutting dentin, selecting the proper bur type is important for improving the bond strength of some self-etching adhesive systems.**

## INTRODUCTION

The smear layer has been defined as a layer of debris on the surface of dental tissues created by cutting a tooth (Eick & others, 1970). It varies in thickness, roughness, density and degree of attachment to the underlying tooth structure according to the surface preparation (Charbeneau, Peyton & Anthony, 1957; Gilboe & others, 1980). As part of restorative procedures in adhesive dentistry, the smear layer must be removed, modified or impregnated by the resin to allow for bonding between the tooth and the restorative material (Swift Jr, Perdigão & Heymann, 1995; Pashley & Carvalho, 1997).

The poor performance of early dentin adhesive systems was thought to occur because the smear layer was not removed, resulting in the adhesive bonding to the surface of the smeared debris (Watanabe, Nakabayashi & Pashley, 1994a) and not to the underlying dentin (Eick & others, 1970). The potential of the smear layer to create an adverse effect on dentin bonding has been reported by Prati and others (1990). The smear layer adheres weakly to dentin, and its removal by an acid demineralizing agent prior to the application of a bonding system has been reported to result in stronger bonds (Pashley, 1991). However, others have reported that the treatment of dentin with acids can cause the collapse of exposed collagen fibers due to the removal of the supporting hydroxyapatite and the denaturation of collagen (Nakabayashi, 1992; Pashley & others, 1993). The remaining matted collagen surface becomes more difficult to impregnate with adhesive monomers. To overcome this problem, the application of a primer aims to restore the permeability of acid-treated dentin and facilitates the penetration of applied monomers. Therefore, the use of an acidic conditioner is necessary to dissolve and remove the smear layer to expose the intertubular and peritubular dentin, remove the debris from the dentin surface and demineralize the superficial dentin matrix, thus allowing the subsequent infiltration of the resin into the dentin surface.

Clinically, after carious dentin has been removed or any other kind of dentin instrumentation has been performed, a smear layer is formed over the dentin surface (Pashley & Carvalho, 1997; Ogata & others, 2001). The nature of the smear layer depends on the type of bur used. In addition, different speeds of the bur and the pressure applied may influence the kind of smear layer.

Coarse and superfine diamond burs each create a different smear layer. Coarse diamond burs create a thick smear layer containing cut collagen fibers and hydroxyapatite crystallites (Ayad, Rosenstiel & Hassan, 1996; Gwinnett, 1984). This can interfere with bonding of the adhesive agents, as it is not easy for some adhesive monomers to permeate dentin smears and impregnate the underlying dentin (Nakabayashi & Saimi, 1996). Differences in the smear layer generated by burs and abrasive papers have been reported to affect the bond strengths of resins to dentin (Tagami & others, 1991; Watanabe, Saimi & Nakabayashi, 1994b).

The "all-in-one" adhesive systems are simple to use, as the steps of etching, priming and bonding occur in one single application step. A previous study of these systems has demonstrated the influence of the type of smear layer generated on their bond strength (Inoue & others, 2001). However, that study found that use of a coarse diamond bur may reduce the possibility of penetration of bonding monomers into dentinal substrate in these systems. It is not known whether these differences are also true for the newer, all-in-one adhesives. The reasons for using self-etching primers include easy handling by the operator (Sano & others, 1998; Miyazaki, Onose & Moore, 2000) and high clinical performance (Latta & others, 1997). These primers were marketed in the last decade and since then, there have been many self-etching primers available. Current advances in these systems are toward one-bottle, all-in-one adhesives. Older versions of self-etching primers were believed to be susceptible to the presence of the smear layer in terms of bond strengths (Toida, Watanabe & Nakabayashi, 1995). When self-etching primers are applied to the smear layer situated on the tooth surface, the acid primer can simultaneously modify or dissolve the smear layer and decalcify the dentin (Watanabe & others, 1994a); this procedure produces good adhesion both to enamel and dentin (Kanemura, Sano & Tagami, 1999). Recent reports have demonstrated that some all-in-one systems bond well to thinner smear layer covered dentin (Inoue & others, 2001; Koase & others, 2004).

This study compared the microtensile bond strength of four adhesive systems (including three all-in-one systems) after preparing the dentinal surface with either a coarse or superfine diamond bur. The null hypothesis was that there are no differences in microtensile bond strengths to dentin prepared with either a coarse diamond bur or a superfine bur.

## METHODS AND MATERIALS

### Adhesives and Bonding Procedures

Twenty-four caries-free extracted human molars, stored at 37°C in distilled water, were used for this study, in accordance with local institutional guidelines. The coronal surfaces of the teeth were trimmed using a

model trimmer (MT-7 Morita Corp, Kyoto, Japan) in order to form a long, flat dentin surface at the mid-crown level. A smear layer was then created by removing a thin layer of the surface with a high-speed diamond bur under water-cooling. Twelve teeth were prepared with a regular-grit diamond bur (Diamond Point FG, #106RD, Shofu, Kyoto, Japan), while the remainder were prepared using a superfine-grit diamond bur (Diamond Point FG, #SF 106RD, Shofu Inc). These two types of dentin substrates were randomly assigned to one of the four bonding treatments carried out according to the respective manufacturers' instructions.

After applying the adhesive in each tooth, resin composite (Clearfil AP-X, Kuraray, Okayama, Japan) was built-up incrementally (in three increments) to a height of 5 mm. Each increment was light cured for 40 seconds, and the specimens were then stored in distilled water for 24 hours at 37°C.

### Microtensile Bond Strength Testing

The specimens were sectioned into six slabs, approximately 0.7-mm thick, perpendicular to the bonded surface using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water. These slabs were trimmed to an hourglass shape to form a gentle curve along the adhesive interface from both sides, using a superfine-grit diamond bur as described by Sano and others (1994). The width at the narrowest portion was approximately 1.4 mm, and the thickness of the bonded area of each specimen was verified by a digital micrometer. The specimens were then attached to a Ciucchi's jig (Paul & others, 1999) with cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Otagawa, Japan) connected to a desktop testing apparatus (EZ test, Shimadzu, Kyoto, Japan).

The specimens were then subjected to a microtensile strength test at a crosshead speed of 1 mm/minute until failure occurred. The tensile bond strength was calculated as the load at failure divided by the bonded area. Bond strength data were analyzed by two-way ANOVA and the Games-Howell test for interaction between the adhesive and type of cut dentin. The surfaces of the fractured specimens were visually inspected under a light microscope (20x), then observed microscopically with SEM to determine the failure mode.

### Failure Mode Analysis

For determining the modes of fracture, both the dentin and composite halves of the fractured

specimens were observed with a FE-SEM microscope (Hitachi S4000, Tokyo, Japan). The failure modes were classified as interfacial (fracture between the dentin or the hybrid layer and the overlying adhesive in the same sample), mixed (interfacial and partial cohesive failure in dentin or composite in the same sample) or cohesive (failure within dentin only, cohesive in adhesive only or cohesive in resin composite only), wherever relevant.

### SEM Observation of Dentin Surface Treated with the Adhesives

In order to understand the effect of conditioning on the dentinal surfaces treated with the two types of burs, a further evaluation was conducted. The coronal surfaces of eight additional teeth were trimmed at the mid-crown level; the flat dentin surfaces of four teeth were then treated using the two burs as previously described in the method. One surface in each group of four teeth was treated with the self-etching primer (CSE, Kuraray, Osaka, Japan) or with one of the all-in-one adhesives (GB, GC Company, Tokyo, Japan; SSB, Kuraray and PLP, 3M ESPE, St Paul, MN, USA) without light curing. The teeth were immediately soaked in 100% acetone for one minute to remove the applied adhesive. All the specimens were dehydrated using an ascending concentration of ethanol and chemical-dried with HMDS, which is a protocol for SEM examination described by Perdigão and others (1995).

## RESULTS

There were no pre-testing failures in the various material groups except for PLP. For this adhesive, four samples in the regular-grit group failed during specimen fabrication, while 11 failed in the superfine diamond group. The means of MTBS and the standard deviation for each group are summarized in Table 1. The CSE group treated with a superfine bur tended to show the highest bond strength ( $65.16 \pm 13.96$  MPa), while PLP treated with a superfine bur tended to produce the lowest value ( $7.60 \pm 9.73$  MPa).

The two-way ANOVA indicated that there was significant interaction between the adhesive and diamond bur surface preparation ( $p < 0.05$ ). Therefore, further

Table 1: Microtensile Bond Strength (MPa) for All Specimen Groups

| Adhesive               | Diamond Bur    |    |    |                |    |    |
|------------------------|----------------|----|----|----------------|----|----|
|                        | Regular-grit   |    |    | Superfine-grit |    |    |
|                        | Mean (SD)      | N  | PF | Mean (SD)      | N  | PF |
| Clearfil SE Bond (CSE) | 49.04 (13.43)* | 20 | -  | 65.16 (13.96)* | 20 | -  |
| G-Bond (GB)            | 33.21 (9.24)   | 18 | -  | 27.00 (6.40)   | 16 | -  |
| SSB-200 (SSB)          | 32.20 (6.83)** | 21 | -  | 40.08 (4.98)** | 23 | -  |
| Prompt L-Pop (PLP)     | 15.28 (8.67)   | 20 | 4  | 7.60 (9.73)    | 19 | 11 |

Values marked with either \* or with \*\* were significantly different from each other ( $p < 0.05$ )  
 N= total number of specimens including the pre-testing failures  
 PF= pre-testing failure

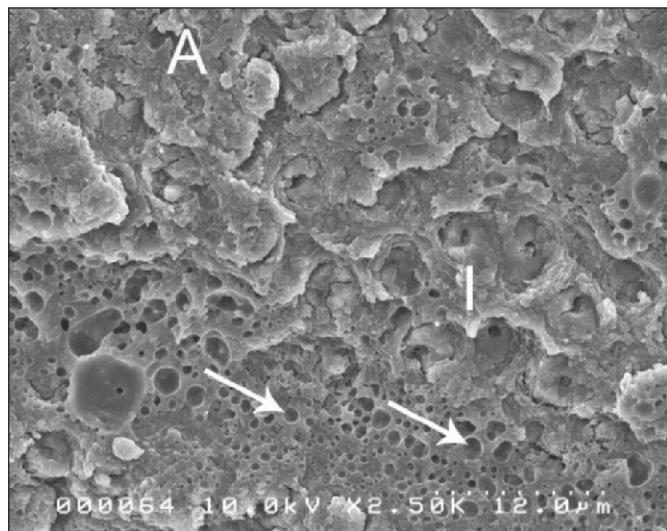


Figure 1. SEM photomicrograph of a typical (both regular and superfine-grit) specimen of GB showing interfacial fracture (I) between the hybrid layer and the adhesive, as well as areas where the failure occurred within planes in the adhesive (A). In addition, "blisters" (arrowed) can be seen in the adhesive.

analysis for significant differences between groups was carried out using the Games-Howell test.

Significant differences were found between MTBS to dentin cut with a regular-grit diamond (lower values) bur and dentin cut with a superfine-grit diamond bur only for the CSE and SSB adhesives ( $p < 0.05$ ). For both GB and PLP, no significant differences in MTBS values were shown between the specimens prepared with regular-grit burs compared to those prepared with superfine-grit burs.

Fractographical analysis of the specimens using SEM revealed a mixed mode of failure in all groups except PLP. The specimens of CSE bonded to dentin cut with the regular-grit diamond bur showed failures at different levels of the adhesive in the same sample, that is, interfacial between the hybrid layer and the adhesive, within the adhesive as well as the interfacial between the adhesive and the overlying resin composite. In the CSE group bonded to dentin cut with the superfine-grit diamond bur, the mixed fractures occurred not only within the adhesive, but they were also interfacial between the dentin and the adhesive.

For the GB group, the mixed fractures for both specimens cut with a superfine and a regular-grit diamond bur were interfacial (between the hybrid layer and the adhesive) and also occurred at different planes in the adhesive within the same sample; the adhesive also showed the presence of "blisters" (Figure 1) in both groups.

In the SSB samples cut with a regular diamond, the mixed failures in each sample were both interfacial between the hybrid layer and the adhesive, as well as

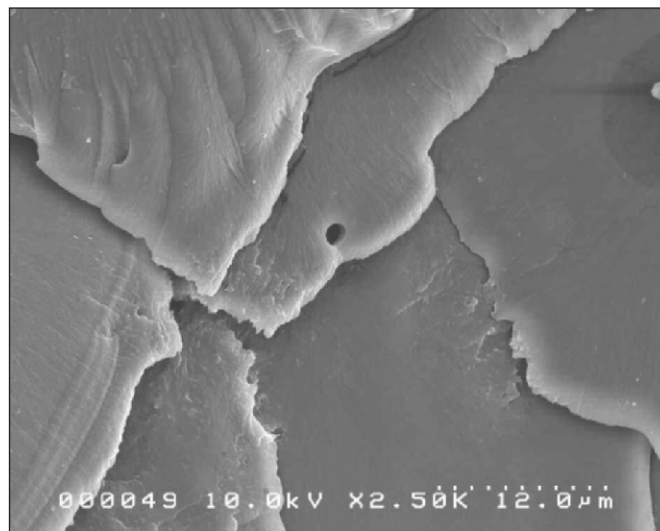


Figure 2. SEM image of a typical PLP (of both regular and superfine-grit) sample showing a cohesive fracture that occurred within the adhesive layer.

within the adhesive. Those prepared with a superfine-grit bur displayed fractures at different planes within the adhesive—cohesive failure within the adhesive.

In the PLP samples treated with both types of burs, all the samples showed failures that were cohesive within the adhesive only (Figure 2).

SEM observations of the conditioned dentin surfaces with the two types of burs revealed differences with the various adhesives. Dentin prepared with regular-grit burs treated with CSE showed partial removal of the smear layer and the peritubular dentin appeared to be slightly etched (Figure 3A). Dentin prepared with superfine-grit burs displayed partial demineralization of the smear layer. The peritubular dentin was also comparatively more etched, and the porosity of the intertubular dentin was slightly greater than the regular grit prepared dentin (Figure 3B).

There were no distinct differences in terms of removal of the smear layer between the dentin cut with a regular-grit bur and dentin cut with a superfine diamond for GB. The smear layer was partially demineralized and the remnants were attached to the dentin surface. The only distinct differences were dissolution of the smear plugs: the smear plugs in the regular-grit prepared dentin were more resistant to the acidity of the adhesive and therefore remained.

For SSB, removal of the smear layer was minimal and similar for both specimens cut with a regular-grit bur and those cut with a superfine diamond. The porosity of the intertubular dentin cut with a superfine-grit bur appeared to be relatively greater than that of regular-grit burs.

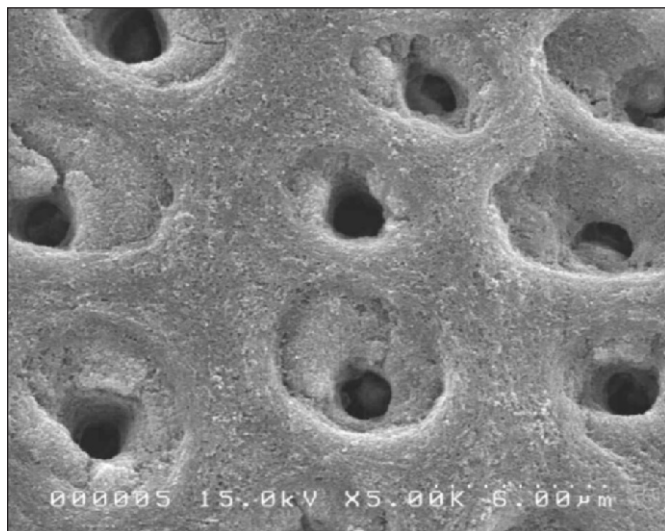


Figure 3A. SEM image of a dentin surface cut with a regular-grit diamond bur, conditioned with CSE, which was not light-cured, followed by rinsing with acetone for 1 minute. The micrograph shows partial removal of the smear layer and the peritubular dentin appears to be slightly etched.

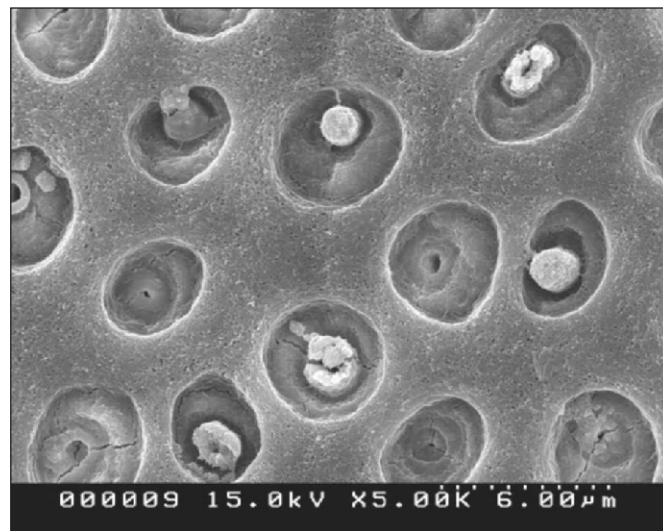


Figure 3B. SEM micrograph of a similarly treated sample as in Figure 3A, but with the dentin cut with a superfine diamond bur. Partial demineralization of the smear layer is noted. The peritubular dentin appears comparatively more etched, and the porosity of the intertubular dentin is slightly greater than that of regular-grit prepared dentin.

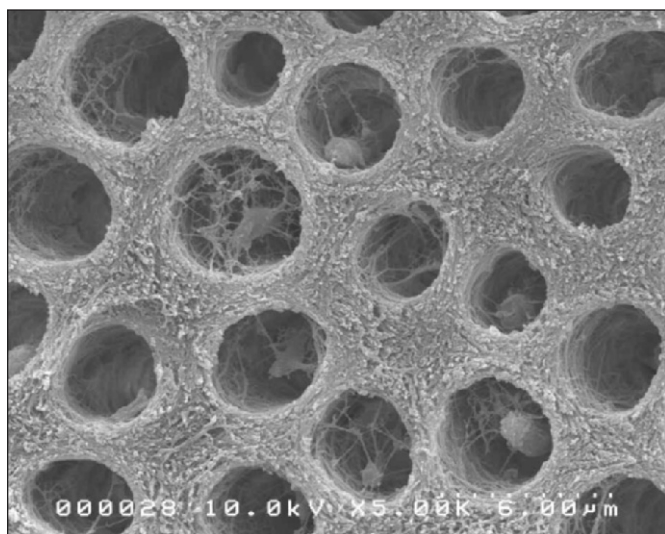


Figure 4A. SEM appearance of a dentin surface cut with a regular-grit diamond bur and conditioned with PLP, which was not light-cured, followed by rinsing with acetone for 1 minute. This shows complete removal of the smear layer and plugs. In addition, the complete dissolution of peritubular dentin can be seen.

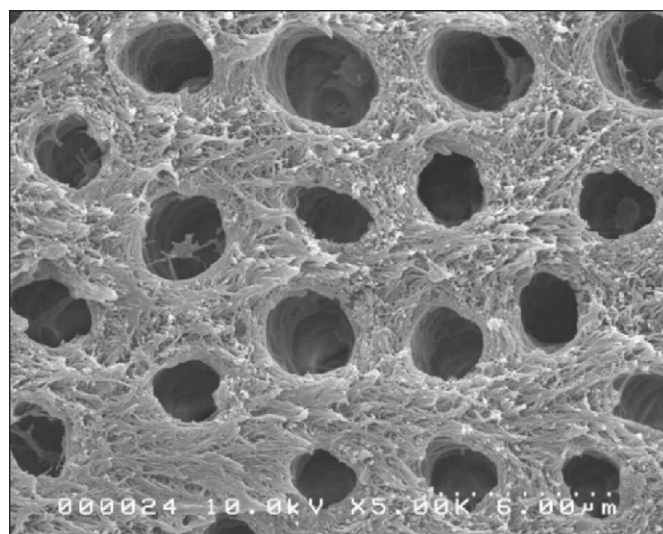


Figure 4B. SEM micrograph of a dentin surface cut with a superfine-grit diamond bur and conditioned with PLP, which was not light-cured, followed by rinsing with acetone for 1 minute. This shows complete removal of the smear layer and plugs, as well as complete dissolution of peritubular dentin as in Figure 4A. The intertubular dentin appears more aggressively etched compared to that cut with a regular-grit bur in Figure 4A.

The PLP samples cut with both a regular-grit and a superfine-grit bur showed complete removal of the smear layer and plugs. Additionally, complete dissolution of peritubular dentin was noted in both. The intertubular dentin cut with the regular-grit bur appeared less aggressively etched compared to dentin cut with the superfine-grit bur (Figures 4A and 4B).

## DISCUSSION

This study assessed the effect of different smear layers generated by two different types of burs on the microtensile bond strengths to dentin.

CSE performed significantly better with superfine cut dentin. CSE primer partially dissolves the smear layer as seen in Figures 3A and 3B. It was previously reported

that a hybridized smear layer and hybridized dentin were observed when using CSE (Tay & others, 2000). CSE can penetrate the partially demineralized smear layer and create a hybrid layer (Tay & others, 2000). The greater porosity of intertubular dentin observed in this study with the superfine-grit preparation implies more channels of penetration of adhesive monomers. This could be the reason for the greater bond strengths achieved with the superfine diamond bur.

For GB, there were no differences in bond strengths between the dentin cut with a regular-grit bur and dentin cut with a superfine diamond bur. This may be attributed to the similar mild removal of the smear layer in both. Another reason there were no differences in bond strengths may be the presence of "blisters" within the adhesive resin. Blisters may create defects within the adhesive resin during tension and initiate the propagation of cracks within the adhesive. The elimination of blisters should be important for producing good bonding between the resin and dentin. Further work is required to determine why blisters occur.

SSB showed significantly higher bond strengths to superfine cut dentin compared to regular cut dentin. Although the smear layer removal for both groups is minimal, increased porosity within the intertubular dentin was found for the superfine grit. This may explain the higher bond strengths observed in this group. Further work, such as using TEM, is necessary to clarify the interaction between the resin and dentin of this system.

PLP was the only adhesive tested which had pre-testing failures in both groups. In addition, PLP tended to show the lowest bond strengths for both groups and consistently demonstrated cohesive failure within the adhesive. Although Figures 4A and 4B show complete dissolution of the smear layer (due to the low pH of 0.8) to expose the collagen fibrils, whether or not the subsequent penetration of resin monomers into the exposed collagen web occurs is not clear. The authors speculate that the relatively lower bond strengths with this adhesive suggest incomplete hybridization. Future studies should focus on the quality of the resin-dentin interface of this system.

## CONCLUSIONS

This study showed that the type of bur used to prepare the dentinal surface may affect microtensile bond strength when using some of the current all-in-one systems. Hence, the null hypothesis that there are no differences in microtensile bond strengths to dentin prepared with either a coarse diamond bur or a superfine bur was rejected. In conclusion, when cutting dentin, selecting the proper bur type is important for improving the bond strength of some self-etching adhesive systems.

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