# Influence of Silver Diamine Fluoride Treatment on the Microtensile Bond Strength of Glass Ionomer Cement to Sound and Carious Dentin

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#### Clinical Relevance

This study provides valuable information about the influence of silver diamine fluoride (SDF) treatment on the microtensile bond strength of glass ionomer cement (GIC) to dentin.

### **SUMMARY**

Objectives: To investigate the influence of silver diamine fluoride (SDF) treatment on the microtensile bond strength (mTBS) of glass ionomer cement (GIC) to sound and artificial carious dentin.

Methods: Thirty dentin blocks prepared from 30 noncarious human molars were randomly allocated into either the sound (Gp1) or artifi-

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cial carious dentin (Gp2) groups. A microbiological method was adopted to create artificial dentin caries lesions in Gp2 specimens. Each dentin block was sectioned into two halves perpendicularly, and each pair of block halves was randomly assigned to two subgroups to receive topical application of SDF (Gp1-SDF, Gp2-SDF) or water as control (Gp1-water, Gp2-water). An encapsulated GIC was bonded to the exposed dentin surfaces 14 days after the SDF/water application. After immersion for 7 days in artificial saliva, the GIC-dentin specimens were sectioned into beams for mTBS testing. Failure mode was examined after the mTBS test.

Results: There was no significant difference in the mean mTBS values between the SDF and control subgroups (Gp1-SDF vs Gp1-water,  $10.57\pm1.6$  MPa vs  $10.20\pm1.8$  MPa; Gp2-SDF vs Gp2-water,  $6.14\pm2.2$  MPa vs  $5.97\pm2.3$  MPa; paired t-test, p>0.05). However, the mean mTBS value of the sound dentin group was significantly higher than that of the carious dentin group, irrespective of whether SDF was applied prior to GIC bonding (independent t-

E272 Operative Dentistry

test, p<0.001). Proportionally more cohesive failures occurred in the sound dentin groups (Gp1-SDF, 48.4%; Gp1-water, 42.9%) compared with the carious dentin groups (Gp2-SDF, 15.6%; Gp2-water, 9.8%; p<0.05).

Conclusions: SDF treatment had no significant influence on the mTBS of GIC to dentin. Compared with sound dentin, dentin with caries had lower mTBS to GIC.

#### INTRODUCTION

Dental caries is a multifactorial disease involving interactions between the tooth structure, the microbial biofilm formed on tooth surface, and sugar intake. Pathological factors can cause an imbalance between demineralization and remineralization, leading to a net loss of minerals from dental hard tissues and ultimately progressing into a carious lesion. Complete removal of all carious dental tissues followed by placement of a restoration in the cavity has been considered the standard procedure for dental caries treatment. However, this approach has been challenged, as modern understanding of dental caries is that caries is not an infectious disease to be cured by removing all bacteria through extended cavity preparation. It is recommended to preserve not only sound dental tissues but also tissues with potential to remineralize, thus maximizing the healing potential of the tooth.<sup>2,3</sup> Indeed, nonrestorative treatments, even with no removal of carious tissue, are options for the management of dental caries.<sup>4,5</sup>

As a nonrestorative caries management method, topical application of silver diamine fluoride (SDF) solution has been shown to be effective in arresting dentin caries in young children<sup>6-9</sup> as well as in older adults. 10 SDF solution is a colorless solution containing diamine-silver ions and fluoride ions. The diamine-silver ion is a complex with two ammonia molecules attached to a silver ion, which makes it more stable and less oxidizing than the silver ion. 11 Silver can inhibit bacterial growth and dentin collagen degradation. The fluoride ion is known to promote remineralization of dental tissues by forming a mixture of fluorapatite and fluorohydroxyapatite on the tooth surface. The combination of silver and fluoride in an alkaline solution has a synergistic effect in arresting active dentin caries, which makes SDF different from other topical fluoride agents. 12 Hence, SDF solution not only acts as an anticariogenic agent to reduce the growth of cariogenic biofilm<sup>13</sup> but also helps carious dentin to remineralize and results in reduced solubility by transforming some hydroxyapatite into fluorohydroxyapatite. 14,15

Although application of SDF can arrest active caries effectively, there are limitations when only SDF treatment is used. Since SDF application is a nonrestorative approach, the cavities in teeth are still present after the application. This might cause problems in oral hygiene effectiveness because food debris and dental plaque can accumulate in the unrestored cavities. Furthermore, the chewing function of broken-down teeth might be compromised. In addition, a drawback of SDF application is the black stain on the arrested caries lesions, <sup>16</sup> which may give rise to esthetic concerns. It was reported that placement of restorations in the SDFtreated caries lesions to cover the black stain increased parental satisfaction with their child's dental appearance. 17 Hence, restoration placement after SDF application can aid plaque control, restore chewing function, and improve dental appearance.

Atraumatic restorative treatment (ART) developed in the 1980s is a minimally invasive operative approach for managing cavitated dentin carious lesions. The ART procedures require removal of soft carious dental tissues using hand instruments without administration of local anesthesia. The cavity is then cleaned and restored with adhesive dental materials, normally high-viscosity glass ionomer cement (GIC), and the adjacent pits and fissures are sealed concurrently. 18 Single-surface ART restorations in both primary and permanent teeth have a high retention/survival rate and are recommended for use in clinical practice. 19 Thus, the ART approach can be adopted to restore SDF-treated dentin caries lesions to reshape the tooth contour and to cover the black stain so as to improve both tooth function and esthetics. It is important to determine whether SDF treatment of the dentin surface influences the bond strength of dentin to GIC.

Previous studies achieved varying results regarding the effect of SDF application on the bond strength of dentin to GIC. 20-24 Nonstandard protocols to prepare specimens can lead to contradictory results. 25 Some of these laboratory studies adopted methods of SDF application that were not recommended or practical in a clinical situation, such as rinsing the SDF away immediately after application or leaving it to air-dry on the dentin surface for a very long period of time. 22 In addition, the test dental substrates varied between studies, including sound, demineralized, and/or natural carious dentin.

It was reported that the mean microtensile bond strength (mTBS) value of GIC to demineralized dentin was significantly lower than that to sound dentin. <sup>26</sup> However, this was not supported by other studies, <sup>22,27,28</sup> in which similar bond strengths were found irrespective of whether the substrate was carious or sound dentin.

In this study, specimens were prepared in ways that simulated clinical situations. The aim was to investigate the influence of SDF treatment on the bond strength of GIC to both sound and artificial carious dentin. Two null hypotheses were tested: 1) SDF treatment has no effect on the bond strength of GIC to sound and carious dentin and 2) there is no difference in the bond strength to GIC between sound and carious dentin.

# **METHODS AND MATERIALS**

# **Specimen Preparation**

The specimen preparation process is shown in Figure 1. Thirty extracted noncarious human molars stored in 0.5% thymol solution before use were selected from the tooth collection of a dental teaching hospital. A low-speed saw (ISOMET 1000, Buehler, LakeBluff, IL, USA) with a diamond blade was used to remove the occlusal, mesial, distal, buccal, and lingual enamel as well as the roots of all 30 teeth under running deionized water to create dentin blocks of approximately 6 mm  $\times$  4 mm  $\times$  6 mm each. The blocks were examined visually with magnification to ensure that there was no enamel on the surface. The dentin blocks were randomly allocated into two groups: the sound dentin group (Gp1) and the artificial carious dentin group (Gp2). With the occlusal surface facing up, each dentin block was sectioned into two halves perpendicularly using the same saw mentioned above. Each pair of tooth halves in both Gp1 and Gp2 was randomly assigned to two subgroups, SDF (Gp1-SDF, Gp2-SDF) and control (Gp1-water, Gp2-water) subgroups, with 15 dentin blocks in each subgroup. The exposed pulp chamber of each dentin block was cleaned with an excavator and then filled with resin composite (DUO-LINK, Bisco, Schaumburg, IL, USA) to act as support. The nonocclusal dentin surfaces were coated with a layer of acid-resistant nail varnish (Clarins, Paris, France) to prevent demineralization in the following artificial caries development procedure. The occlusal dentin surface was polished with a 2000-grit silicon carbide paper under running deionized water to create a fresh and uniform dentin surface.

In this study, 5 common species of cariogenic bacteria—Streptococcus mutans ATCC (American Type Culture Collection) 35668, Streptococcus sobrinus ATCC 33478, Lactobacillus acidophilus ATCC 9224, Lactobacillus rhamnosus ATCC 10863, and Actinomyces naeslundii ATCC 12014—were chosen to create artificial caries lesions in the dentin blocks. Five equal aliquots of 10<sup>7</sup> CFU/mL of each bacterium in brain-heart infusion broth were mixed and inoculated on the exposed surface of the 30 dentin blocks in Gp2. Afterward, each dentin block was put into 1 mL of 5% sucrose solution in one well of a 24-well plate and incubated anaerobically at 37°C for three days.<sup>29</sup>

After rinsing with water, the exposed surface of the dentin block was dried with cotton pellets. Then, 38% SDF solution (Saforide, Toyo Seiyaku Kasei Co Ltd, Osaka, Japan) was applied onto the exposed dentin surface of the dentin blocks in the SDF subgroups (Gp1-SDF and Gp2-SDF) with a microbrush (microapplicator, regular, Premium Plus International Ltd, Hong Kong) for 10 seconds and remained undisturbed for about 15 seconds. Dentin blocks in the control subgroups (Gp1-water and Gp2-water) received deionized water following the same procedure described above. After the application of SDF/water, the dentin blocks were immersed into artificial saliva for 14 days before bonding to GIC. The artificial saliva was refreshed every 24 hours.

Silicone impression material (DMG, Chemisch-Pharmazeutische Fabrik GmbH, Hamburg, Germany) was used to create a mold to help bond the GIC to the exposed dentin surface. Before bonding, the dentin surface was cleaned by deionized water and then dried with cotton pellets. The surface was then conditioned by the polyacrylic acid of a GIC product (Ketac-Molar, 3M ESPE Dental Products, St Paul, MN, USA) for 10 seconds. The surface was cleaned for one to two seconds with cotton pellets dipped in water and then blot dried. An encapsulated GIC material (Ketac-Molar Aplicap, 3M ESPE Dental Products) was bonded to the exposed dentin surface following the manufacturer's instructions, that is, mixed in a high-frequency electric-driven mixing device for 10 seconds and then placed onto the dentin surface with an applicator. After setting for two minutes, the GIC-dentin specimen was removed from the mold. The GIC-dentin specimen was then coated with a layer of petroleum jelly (Vaseline, Unilever, Englewood Cliffs, NJ, USA) to mimic the recommended clinical practice in ART

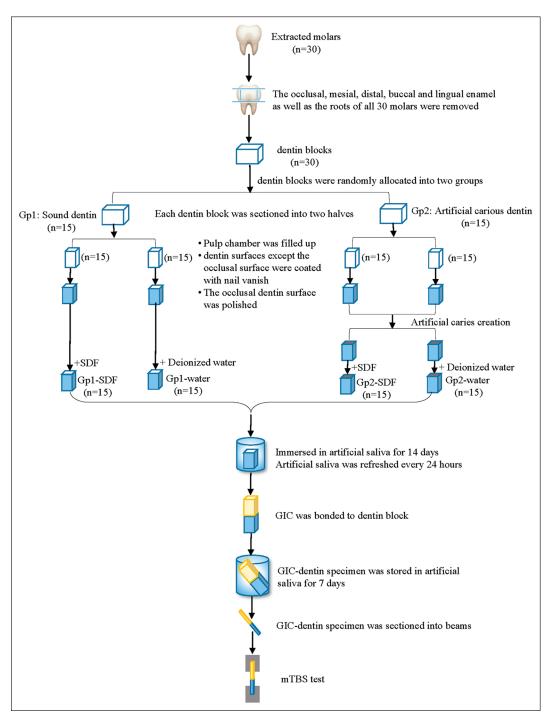


Figure 1. Schematic of sample preparation.

approach and stored in artificial saliva for seven days before mTBS testing.

# mTBS Test

The GIC-dentin specimens were sectioned into beams perpendicular to the interface surface that were approximately  $1 \text{ mm} \times 1 \text{ mm}$  in cross section.

For each GIC-dentin specimen, three to five beams were successfully obtained. The length and width of the bonded surface area were measured by a digital caliper (Mitutoyo, Kawasaki, Japan). The mTBS of each beam was tested by a universal testing machine (model 4444, Instron, Norwood, MA, USA) using a cyanoacrylate glue (Zapit, Dental Ventures of Amer-

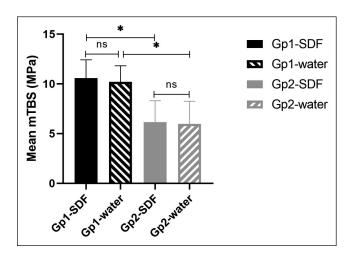


Figure 2. Mean mTBS values of the four study subgroups; ns, no significant difference (paired t-test); \*, p<0.001 (independent sample t-test).

ica, Corona, CA, USA) with a 100-N load cell at a crosshead speed of 1 mm/min. The mTBS value (MPa) of each beam was calculated by dividing the breaking peak value (N) by the bonded surface area (mm<sup>2</sup>).

# **Failure Mode Analysis**

Failure mode of the bonded surface was examined with a scanning electron microscope (SEM; SUI5IO, Hitachi, Japan). The specimens were coated with 80% Pt and 20% Pd for 2.5 minutes before scanning. Failure modes were classified into three types: 1) adhesive failure at the dentin and GIC material interface, 2) cohesive failure within the GIC material or dentin, and 3) mixed failure with a combination of both types of failures.

### **Data Analysis**

Since the beams from the same GIC-dentin specimen cannot be considered as independent samples, the mean mTBS value of all successfully sectioned beams from the same GIC-dentin specimen was used as the mTBS value of that specimen.

All statistical analyses were conducted using statistical software (SPSS, Versin25, IBM Corp, Armonk, NY, USA). Paired *t*-tests were used to assess the mTBS values of paired specimens in the SDF and control subgroups. Independent sample *t*-tests were conducted to compare the mTBS values of the specimens in the sound and carious dentin groups. In addition, the distribution of failure modes in the four study subgroups was compared by chisquare testing with multiple comparisons using the Bonferroni method. The statistical significance of all tests was set at 0.05.

#### **RESULTS**

The mean area of the bonded surface was  $1.087 \pm 0.159 \text{ mm}^2$ , with no significant differences among the four study subgroups (p>0.05). The mean mTBS values of Gp1-SDF  $(10.57\pm1.6 \text{ MPa})$  and Gp1-water  $(10.20\pm1.8 \text{ MPa})$  were not significantly different (p>0.05; Figure 2). Similarly, there was no significant difference between the mean mTBS values of Gp2-SDF  $(6.14\pm2.2 \text{ MPa})$  and Gp2-water  $(5.97\pm2.3 \text{ MPa}; p>0.05)$ . However, the mean mTBS values of the sound dentin groups were significantly higher than those of the carious dentin groups, irrespective of whether SDF was applied prior to GIC bonding (p<0.001).

The distribution of failure modes of the four study subgroups is shown in Table 1. Three types of failure modes, adhesive (47.9%), cohesive (30.2%), and mixed (21.9%) failure, were observed. The representative SEM images of the GIC-dentin interfaces are displayed in Figure 3. All cohesive failures occurred in the GIC materials with no dentin surface exposed. Proportionally more cohesive failures were observed in the sound dentin groups (Gp1-SDF, 48.4%; Gp1-water, 42.9%) compared with the carious dentin groups (Gp2-SDF, 15.6%; Gp2-water, 9.8%; p<0.05). Mixed failure mode was more often observed in the Gp2-SDF (34.4%) group compared with the Gp1-SDF group (10.9%; p<0.05). There was no significant

Table 1: Distribution of Failure Modes in the Four Study Subgroups <sup>a</sup>					
Failure Mode	Group, n (%)				Total
	Gp1-SDF*	Gp1-Water*	Gp2-SDF*	Gp2-Water*	
Adhesive	26 (40.6)	26 (41.3)	32 (50.0)	32 (58.8)	116 (47.9)
Cohesive	31 (48.4) д	27 (42.9) A	10 (15.6) в	5 (9.8) в	73 (30.2)
Mixed	7 (10.9) A	10 (15.9) AB	22 (34.4) в	14 (27.5) AB	53 (21.9)
Total	64 (100.0)	63 (100.0)	64 (100.0)	51 (100.0)	242 (100.0)

<sup>&</sup>lt;sup>a</sup> Multiple comparison among the four subgroups was tested by Bonferroni method. Different letters denote that groups in the same failure mode (within the same row) differ significantly at the 0.05 level.

\*p<0.001; the p-value was derived from the chi-square test.

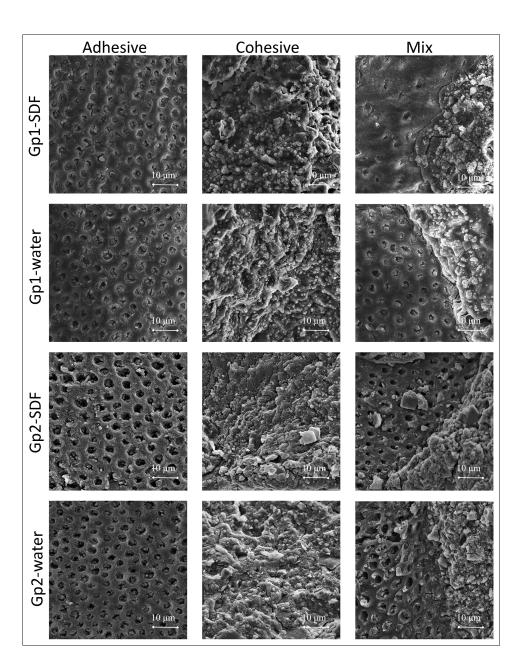


Figure 3. Representative SEM images of failure mode at 1500× magnification

difference in failure mode distribution between the SDF and control subgroups (Gp1-SDF vs Gp1-water, Gp2-SDF vs Gp2-water; p>0.05).

## **DISCUSSION**

In the present study, because the differences in mTBS values between the SDF and control subgroups were not statistically significant, the first null hypothesis could not be rejected. On the contrary, the mTBS of carious dentin to GIC was significantly lower than that of sound dentin to GIC in both the SDF and control subgroups. Therefore, the second null hypothesis was rejected.

A microbiological method using five common species of cariogenic bacteria was adopted to create the artificial dentin caries lesions in the present study. In natural dentin caries lesions, the collagen in the outer carious dentin degrades quantitatively and qualitatively. Compared with those induced by chemical methods, dentin caries lesions created by the microbiological method are morphologically closer to natural caries lesions. Subsequently, the SDF-treated dentin blocks were placed in artificial saliva for 14 days to provide an environment high in calcium and phosphate ions for remineralization. However, it was still different from the complex *in* 

vivo situation. Therefore, caution should be exercised when interpreting the study results.

There was no significant difference in the bond strength between the SDF and control subgroups in the current study. Previous studies reported inconsistent results regarding the effect of SDF application on the bond strength of restorative materials to dentin. Some studies reported that SDF had no significant influence, 20,21,32,33 while others concluded that SDF solution application reduced the bond strength. 23,24,34,35 It is noted that the different studies used different protocols to prepare specimens for testing, and this might be a reason for the inconsistent findings.<sup>25</sup> In clinical practice, SDF is likely to be applied on dentin with a microbrush without immediate rinsing with water or drying with compressed air after the application. Patients are usually instructed not to eat or drink for at least half an hour after SDF application.8 It seems impossible to have the SDF naturally air dried on the dentin surface in the oral cavity since the mouth is always moist with saliva being present. Hence, in the present study, instead of rinsing with water or airdrying immediately after SDF application, the treated dentin blocks were stored in artificial saliva after SDF application to simulate the clinical situation.

Furthermore, different from previous studies in which the SDF-treated dentin surface was polished with a 600-grit silicon carbide paper, 21,34 the SDFtreated dentin surface in the present study was simply cleaned with water without any polishing before GIC bonding. This was done to simulate the clinical practice of the ART procedure in which the SDF-arrested hard dentin surface would not be removed before restoring the cavity with GIC. Ex vivo studies found that there was a significant increase in microhardness with an elevated level of calcium and phosphorus in the outmost surface layer of SDF-arrested dentin caries lesions. 14,36 Meanwhile, fluorohydroxyapatite with reduced solubility formed by the reaction of SDF with calcium and phosphate is considered a main factor favoring remineralization and caries arrest. 15 In clinical practice, as recommended by the minimally invasive approach to maximize the preservation of tooth tissue, no action should be taken to remove the SDF-treated dentin before placement of GIC restorations. Therefore, it is important to keep the SDFtreated dentin surface without any removal and polishing when preparing specimens to assess the bond strength of GIC to SDF-treated dentin.

In the present study, it was found that the bond strength of carious dentin to GIC was significantly lower than that of sound dentin. This finding is in agreement with that of a previous study.<sup>26</sup> The chemical bonding between GIC and dental substrate involves the formation of ionic bonds between the carboxylate functional groups in the polyalkenoic acid molecules and calcium ions in the hydroxyapatite.<sup>37</sup> As caries progresses, demineralization will lead to a loss of calcium ions, and there will be less opportunity for bonding between calcium ions and the carboxyl groups, resulting in reduced bond strength. Therefore, it is expected that the bond strength of GIC to carious dentin is lower than that of GIC to sound dentin.

Three failure modes were observed under SEM in the present study, and the distribution of failure modes was not significantly different between the SDF and control subgroups. In contrast, compared with the artificial caries groups, the sound dentin groups had higher mean mTBS values and more cohesive failures within GIC. This finding is consistent with that of a previous study. The difference in the prominent failure modes between the sound and carious dentin groups is probably related to the considerably lower bond strength of GIC to carious dentin. Nevertheless, cohesive failure is considered as a measure of the tensile strength of the GIC material, rather than the true adhesive bond strength. The strength of the GIC material, and the strength of the GIC material, rather than the true adhesive bond strength.

In the present study, an encapsulated chemically cured GIC was used to test bond strength. Because the powder/liquid ratio influences the physical properties of GIC, 38 an encapsulated GIC instead of a hand-mixed one was used to standardize the preparation of specimen. Despite this, a limitation of this study is that only one commercial brand of conventional chemical-cured GIC was used. Resinmodified GIC (RMGIC) containing a polymerizable monomer, 2-hydroxyethyl methacrylate (HEMA), as an additional component is considered to have a better bonding performance to dental substrates compared with conventional GIC. 26,37,39 The presence of HEMA enhances the micromechanical interlocking between the dentin surface and cement, which leads to a higher bond strength. 40 Therefore, further study is needed to determine whether application of SDF on the dentin surface affects HEMA polymerization, which may lead to a change of bond strength of RMGIC to dentin.

The mTBS test was adopted in the present study to investigate the bond strength of GIC to dentin. The mTBS test has several advantages over the E278 Operative Dentistry

traditional TBS test, such as a greater percentage of adhesive failures and higher possibility of measuring a high bond strength value. 41 In addition, the mTBS test is thought to have greater discriminative power than the traditional shear bond strength (SBS) test.<sup>25</sup> The SBS test is considered to have very little value in the prediction of clinical performance, whereas the mTBS was reported to be associated with the retention rate of Class V restorations in clinical studies. 42 However, a study reported that there was no correlation between bond test results and the retention rate of restorations and only a moderate correlation between mTBS test results and marginal discoloration of restorations. 43 The uncertain correlation between bond strength test results and clinical parameters indicates that one should not solely rely on laboratory bond strength tests. Instead, well-designed clinical trials are needed to determine the performance of restorations in a clinical situation.

#### CONCLUSION

Within the limitations of the present study, it was concluded that SDF treatment had no significant influence on the mTBS of GIC to dentin. However, compared with sound dentin, carious dentin had a lower bond strength to GIC.

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## **Regulatory Statement**

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Hong Kong. The approval code issued for this study is UW 19-253.

#### **Conflict of Interest**

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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