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**Original Research**



# Preliminary Exploration of a Laser-Based Surface Microtexturing Strategy for Improving the Wear Resistance of Dentin: An *In Vitro* Study

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**Abstract**

***Objective:*** Herein, a feasible strategy based on a femtosecond laser (fs-laser) was provided to decelerate dentin wear, and the effect of wear resistance improvement and its potential mechanism were explored.
***Background:*** Tooth wear is a common phenomenon that exists throughout life. While once dentin is exposed, it wears away very quickly. Decelerating tooth wear, especially dentin wear, is an important issue, but there is no ideal treatment.
***Materials and methods:*** Sixteen third molar dentin samples were randomly divided into the amalgam (N1 = 8) and polymer infiltrated ceramic network (PICN) (N2 = 8) groups. One half of the sample was used as the experimental object and the other for the blank self-control. Array microcavities were fabricated on the experimental parts of all the samples with an fs-laser and then filled with the corresponding materials. The experimental and control parts of all the samples were subjected to 3600 cycles of sliding wear tests with titanium balls. The coefficients of friction (COFs) of every friction pair and the temperature rise were recorded in real time. The wear volume and depth were measured by a laser confocal microscopy. Statistical differences of wear volume and depth between the experimental and self-control parts were calculated by paired *t*-test. The wear mode was observed with scanning electron microscopy (SEM).
***Results:*** The COFs in the two experimental groups were significantly decreased. The maximum temperature rise on the dentin surface was 2.0°C. The wear resistance in the amalgam and PICN groups was 4.48 and 3.53 times higher than blank dentin, respectively ($p < 0.001$). The SEM images showed fewer plough grooves and cracks in dentin after microtexturing.
***Conclusions:*** This method could significantly improve tooth wear resistance by reducing the COFs between the friction pairs and reducing the dentin's ploughing effect and provide new treatment ideas for slowing the loss of severely worn dentin.

**Keywords:** tooth wear, wear behavior, femtosecond laser, laser therapy, dentin

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## Introduction

Tooth wear is a common phenomenon that refers to the loss of hard tissue in teeth without caries or trauma.[1,2] Wei et al.[3] found in 2014 that the prevalence of tooth wear in the 35–49 and 50–74 age groups was 67.5% and 100%, respectively. The wear resistance of dentin is lower than that of enamel due to the lack of enamel rods and the reduction of hydroxyapatite content. Once dentin is exposed, it wears away very quickly. At present, according to the European consensus on management guidelines of severe tooth wear[4] in 2017, direct resin filling and full crown restoration have some inevitable disadvantages as classic prosthodontic methods. Full and partial crowns, in addition to being costly, carry a high risk of complications, some of which even compromise the prognosis of teeth, and direct resin filling has a relatively high failure rate.

To delay the loss of dental tissue, in addition to clinical treatments,[4] biomineralization has been a hot topic for the regeneration or strengthening of teeth to protect weak remaining hard tissue. Yamagishi et al. synthesized an enamel-like assembly structure through *in vitro* experiments, but the process was too complicated to apply to clinical practice.[5] Shao et al.[6] produced a precursor layer to induce the epitaxial crystal growth of hydroxyapatite enamel. However, whether this method can deposit hydroxyapatite on dentin has not been reported.

On the contrary, Esteves-Oliveira et al. either used a remineralization fluid alone or combined it with a laser for treatment and greatly improved the erosion resistance of enamel; however, no wear resistance test was performed.[7–13] Anastasiou et al.[14] sintered an antiwear film on enamel by a femtosecond laser (fs-laser) or $CO_2$ laser. However, the temperature in the sintering process is too high to meet the needs of clinical applications.

The goal of decelerating tooth wear and, specifically, dentin wear is an important issue. However, few studies have focused on improving the wear resistance of the tooth surface. Recently, surface microtexturing has been an effective method to improve the friction and wear properties of mechanical surfaces.[15,16] Meanwhile, the fs-laser is an ultrashort pulse laser with a pulse width of $10^{-15}$ sec. Due to its ultrashort pulse duration, the material will be directly carried away by plasma before the heat can spread around, which can be regarded as ''cold'' processing. Studies[17–19] have shown that an fs-laser allows the efficient ablation of dentin with negligible thermal degradation of the remaining tissue.

The purpose of this study was to provide a microtexturing method to improve the wear resistance of teeth with dentin exposed without changing their macroscopic shape and occlusal contact. The null hypothesis was that there is no significant difference between the mean reductions of the microtexturing part and self-blank dentin.

## Materials and Methods

The study was approved by the local Research Ethics Committee (PKUSSIRB-201949124). The flowchart of this experiment is shown in Fig. 1. Microcavities were fabricated on half of the dentin surfaces with an fs-laser and then microfilled with amalgam or polymer infiltrated ceramic network (PICN), leaving another part as a blank control. The ball-on-flat linear reciprocating wear test was performed on both parts of every sample. Every sample was subjected to wear tests on both the experimental half and blank dentin half. Then, we measured the wear volume and depth to determine whether there was a significant improvement between their wear resistance values.

Based on the results of a pre-experiment, sample size calculations were performed using the PASS software package version 11.0, and the sample size was calculated as 8. Several extracted human third molars were collected and stored in 0.1% thymol solution within one week up to the beginning of the study. Sixteen molars of them were cut perpendicular to the longitudinal axis of the tooth and then prepared into dentin samples with a thickness of 2 mm. Then, the dentin samples were embedded in a self-curing resin with their upper surfaces exposed.

An fs-laser (the parameters are shown in Table 1) emitting at a 1 kHz repetition rate was used for irradiation.[20] The processing schematic diagram is shown in Fig. 2. The chosen laser scanning strategy had a meandering form (Fig. 2c) that fits a circle. The parameters we used are also shown in Table 1. Array microcavities with a diameter of $\sim 600\ \mu m$ were fabricated on the experimental side of each dentin sample with a spacing of $200\ \mu m$. A pulp cavity temperature measurement model was prepared, and the crown enamel and 5 mm of the apical portion of an isolated molar were removed, with dentin exposed on the upper surface. One thermocouple head was inserted into the pulp cavity, and another was placed on the dentin surface to measure the rise in temperature during laser processing.

Microcavities in the amalgam and PICN groups were then filled with a high-copper amalgam (Antai, Inc., China) and PICN (Ceramage; Shofu, Japan), respectively. All the samples in the amalgam group were polished after filling and then left for 24 h. Samples in the PICN group were filled and then irradiated and cured with a specific illumination. After 24 h, all samples were again polished to 3000-grit sandpaper with a water aid.

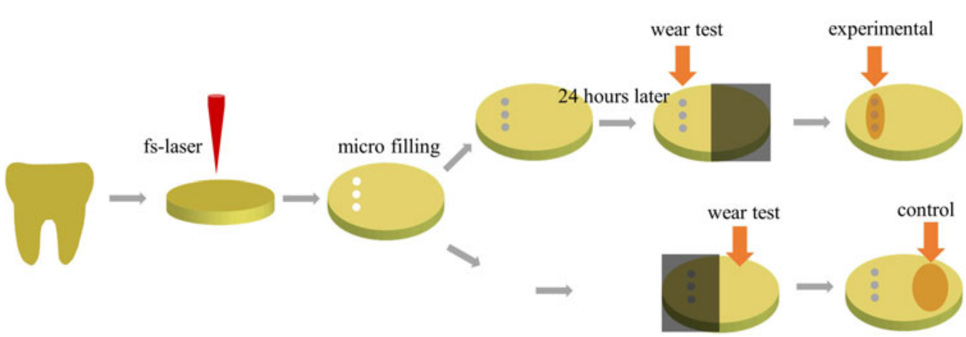


**FIG. 1.** The flowchart of the experiment.

TABLE 1. THE PARAMETERS OF LASER IN THE EXPERIMENT

| *Company* | *Spectra-physics* |
|---|---|
| Wavelength | 800 nm |
| Pulse width | 35 fs |
| Repetition frequency | 1000 Hz |
| Maximum energy per pulse | 3.5 mJ |
| Spot diameter | 21.6 $\mu$m |
| Power | 15 mW |
| Fluence | 10.23 J/cm$^2$ |
| Space of scanning lines | 10 $\mu$m |
| Percentage of spot overlap | 95.37% |
| Percentage of line overlap | 53.70% |
| Scanning speed | 1000 $\mu$m/s |

Tribological tests were conducted in a ball-on-flat configuration with a commercial wear machine (UMT-2). TA2 titanium balls[21] with a diameter of 6 mm served as antagonists. The amalgam and PICN groups and the blank dentin in the two groups served as the substrate. Therefore, combined with the antagonists, the three different types of substrate in the two groups formed three pairs of friction. The three pairs of friction were subjected to a load of 30 N,[22–24] a reciprocating amplitude of 2.5 mm, and a frequency of 2 Hz.[25] All experiments were performed in artificial saliva lubrication[26] at room temperature for 3600 cycles. All dentin samples were subjected to the test twice on both the experimental and control parts. The computer recorded the real-time coefficients of friction (COFs), and the COF curves were plotted in Excel.

The wear volume and depth of the wear scars were obtained with a laser confocal microscope (OLS5000; LEXT; Olympus, Japan). The wear mechanisms were observed using scanning electron microscopy (SEM; SU8010; Hitachi, Japan). To eliminate the individual differences among different teeth, the wear volume and depth of the control part of each specimen were divided by those of the experimental part on the same tooth to obtain a ratio to represent the wear results. The ratios of all dentin samples were calculated.

All of the measured and calculated results were input into the IBM SPSS statistics program (version 24.0), and it was verified that the data obeyed a normal distribution. Paired sample $t$-tests were performed to assess the differences in the wear volume and wear depth of the experimental and control parts in the two groups. A $p$-value of <0.05 was considered statistically significant.

## Results

Temperature changes during the preparation of microcavities with the fs-laser are shown in Fig. 3. In the whole process of array microcavity processing, the maximum temperature rise of the pulp cavity is barely 0.6°C, and the maximum temperature rise of the dentin surface is 2.0°C.

The COF curves obtained in three friction pairs are shown in Fig. 4. The three friction pairs experienced a similar process. In the first few seconds, the COFs increased sharply and then zigzagged upward slowly. The COFs of the dentin are indicated by the yellow line, which finally reached $0.51 \pm 0.04$. After microfilling with amalgam and PICN, the COFs dropped to $0.24 \pm 0.02$ and $0.35 \pm 0.02$, as shown by the red and blue lines, respectively.

The wear volume and wear depth in the two experimental groups were significantly decreased ($p < 0.001$). As shown in Table 2, the wear volume of the dentin was $\sim 4.48 \pm 2.12$ times that of the experimental part in the amalgam group and $3.53 \pm 1.20$ times that in the PICN group. The ratios of the wear depth were $2.39 \pm 0.64$ and $2.12 \pm 0.59$, respectively. The three-dimensional morphologies of the wear scars are shown in Fig. 5.

The SEM images for the three friction pairs are shown in Fig. 6. From left to right, the three columns show the wear mode of dentin at magnifications of 30×, 600×, and 2000× in three friction pairs. The yellow boxed regions are shown at a higher magnification on the right. From top to bottom, three rows indicate blank dentin (Fig. 6a–c), dentin in the wear scar of the PICN group (Fig. 6d–f), and the dentin in the wear scar of the amalgam group (Fig. 6g–i). In the wear scar of blank dentin, at higher magnification (Fig. 6c), several plow marks parallel to the siding direction can be seen, as shown in the red pane. A large area of exfoliation and cracks, as shown by the yellow circle, can be observed in the dentin.

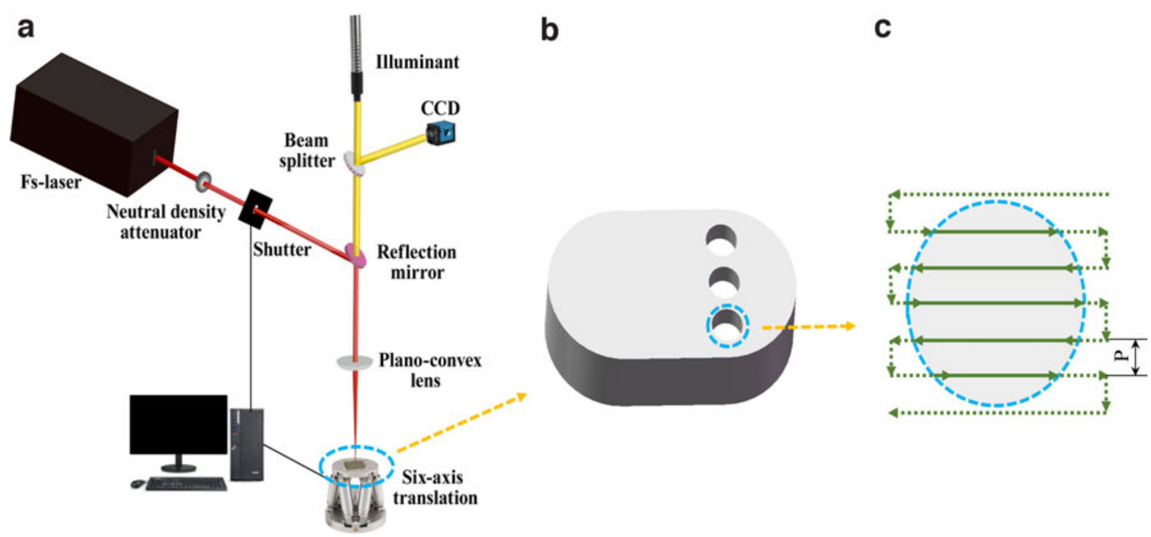


**FIG. 2.** Scheme of the fs-laser used for the ablation of the dentin samples **(a)**. Dentin samples were placed on the translation. The scanning paths of fs-laser for microcavity preparation are shown in **b** and **c**. fs-laser, femtosecond laser.

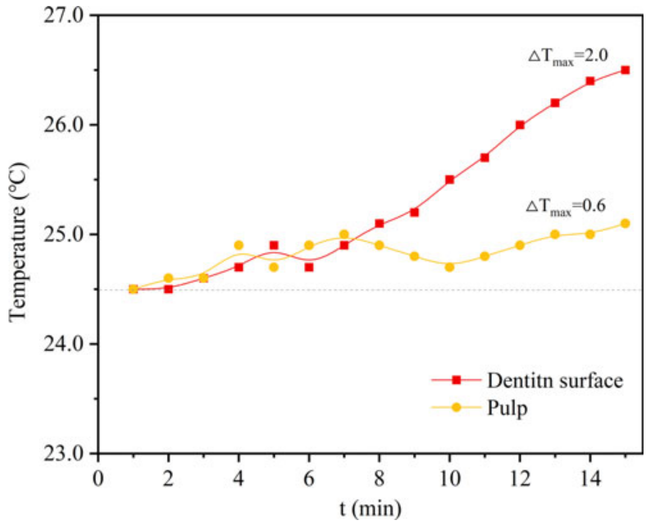


**FIG. 3.** The rise of temperature of dentin surface and pulp during the fs-laser processing.

For the dentin part of the wear scar in the PICN group (Fig. 6f), the range and size of the exfoliation were obviously smaller than those of the blank dentin, and some cracks, as shown by the blue arrow, were visible. In the amalgam group (Fig. 6i), exfoliation and cracks (the blue arrow indicates a tiny crack) could barely be observed in the dentin part; instead, large particles adhered to the surface, as shown by the green arrow. The huge crack in Fig. 6d was caused during the oven-dried procedure overnight before SEM observation.

All the data were normally distributed with a nonsignificant Shapiro–Wilk result ($p > 0.05$). As shown in Table 3, the paired samples $t$-test indicated that the wear volume and wear depth of the experimental and control parts in the two groups were significantly different ($p < 0.001$).

## Discussion

In the present study, we investigated whether this strategy could potentially improve the wear resistance of dentin. To clarify the effect, we performed wear tests on dentin samples, including both the experimental parts and the blank control parts. We detected a reduction by measuring the volume and depth of the wear scars with a laser confocal microscope and

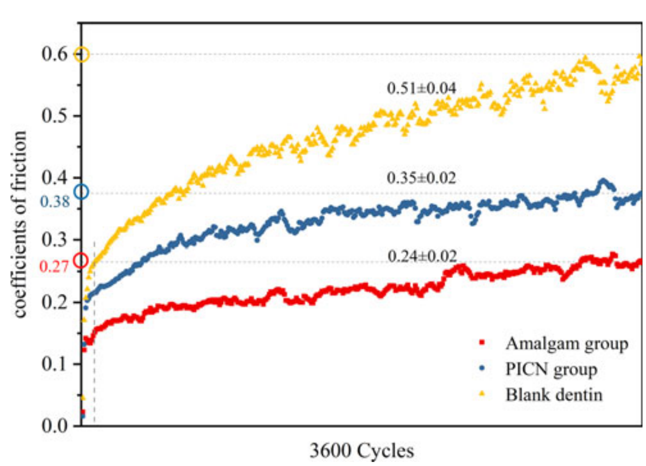


**FIG. 4.** Coefficients of friction during 3600 cycles for the three friction pairs.

Table 2. The Wear Volume and Wear Depth in the Experimental Parts and Self-Control Parts and Their Ratios of the Amalgam Group and Polymer Infiltrated Ceramic Network Group

| Amalgam group | | | | | | PICN group | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Volume ($\mu m^3$) | | | Depth ($\mu m$) | | | Volume ($\mu m^3$) | | | Depth ($\mu m$) | | |
| Amalgam ($A_v$) | Blank dentin ($B_v$) | Ratio ($B_v/A_v$) | Amalgam ($A_d$) | Blank dentin ($B_d$) | Ratio ($B_d/A_d$) | PICN ($P_v$) | Blank dentin ($B_{v2}$) | Ratio ($B_{v2}/P_v$) | PICN ($P_d$) | Blank dentin ($B_{d2}$) | Ratio ($B_{d2}/P_d$) |
| 549596385.38 | 1164683963.01 | 2.12 | 213.57 | 277.76 | 1.30 | 212138625.48 | 1168547670.72 | 5.51 | 75.40 | 254.57 | 3.38 |
| 225855264.49 | 1512336648.21 | 6.70 | 103.02 | 326.46 | 3.17 | 249219967.49 | 1024294776.74 | 4.11 | 107.69 | 280.11 | 2.60 |
| 199481668.53 | 1327580362.04 | 6.66 | 99.06 | 285.48 | 2.88 | 419236207.91 | 947335453.13 | 2.26 | 132.47 | 212.68 | 1.61 |
| 394739442.05 | 1208909398.25 | 3.06 | 135.53 | 312.06 | 2.30 | 399656177.81 | 1198456516.93 | 3.00 | 134.48 | 271.78 | 2.02 |
| 428401275.69 | 1167841188.17 | 2.73 | 81.72 | 269.57 | 3.30 | 323702429.91 | 1643828558.52 | 5.08 | 139.39 | 320.91 | 2.30 |
| 136896200.49 | 1099072532.55 | 8.03 | 133.58 | 306.35 | 2.29 | 572626797.56 | 1575978574.70 | 2.75 | 204.98 | 323.35 | 1.58 |
| 368093309.17 | 1313084465.38 | 3.57 | 173.26 | 335.21 | 1.93 | 317813362.65 | 1118743518.06 | 3.52 | 134.64 | 259.17 | 1.92 |
| 480506625.79 | 1433734162.99 | 2.98 | 145.33 | 285.53 | 1.96 | 522832487.02 | 1051042272.74 | 2.01 | 183.06 | 285.57 | 1.56 |
| Mean ± SD | | 4.48 ± 2.12 | | | 2.39 ± 0.64 | | | 3.53 ± 1.20 | | | 2.12 ± 0.59 |

$A_v/A_d$, the wear volume/depth of the wear scars in the experimental parts of amalgam group; $B_v/B_d$, the wear volume/depth of the wear scars in the self-control parts of amalgam group; $P_v/P_d$, the wear volume/depth of the wear scars in the experimental parts of PICN group; $B_{v2}/B_{d2}$, the wear volume/depth of the wear scars in the self-control parts of PICN group.

PICN, polymer infiltrated ceramic network; SD, standard deviation.

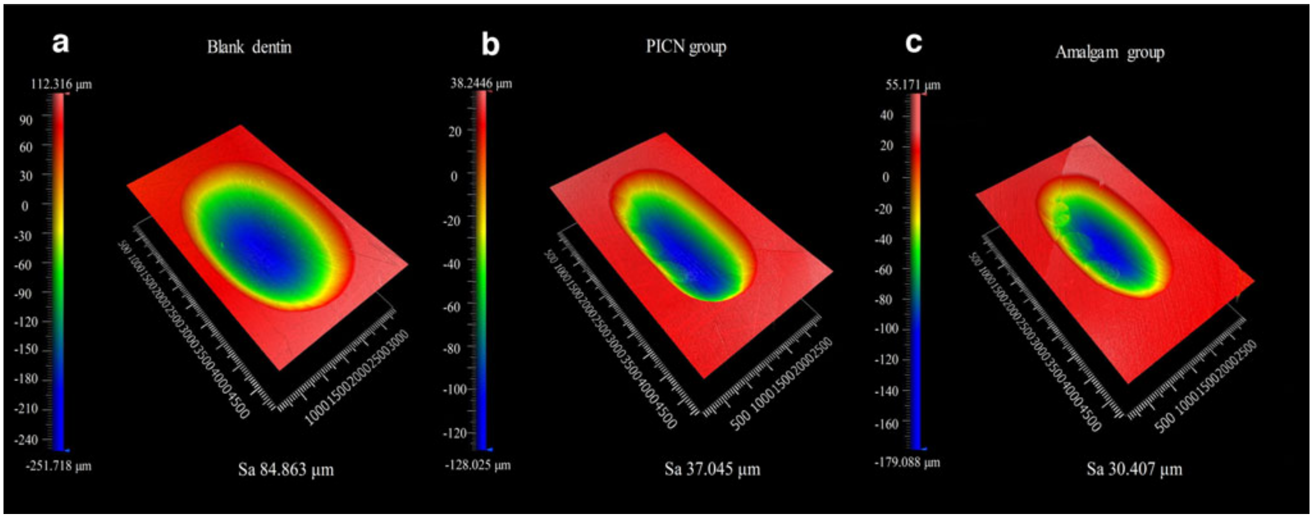


**FIG. 5.** Three-dimensional morphology of wear scars in the blank dentin **(a)**, PICN group **(b)**, and amalgam group **(c)**. PICN, polymer infiltrated ceramic network.

analyzed the wear mechanism with SEM. Our results showed that the wear resistance of the dentin microfilled with prosthetic material was significantly improved. The null hypothesis was rejected. The wear resistance in the amalgam and PICN groups was 4.48 and 3.53 times higher than that of the blank dentin. It can be inferred from the wear depth that after applying this strategy, the time before the pulp in a severely worn tooth is exposed can be doubled.

The ablation threshold[18,27–29] for fs-laser processing of dentin in the literature was $0.3 \sim 1.4\,J/cm^2$, and the fluence used in this experiment was $10.23\,J/cm^2$, which was 7.3–34 times the ablation threshold. Under the experimental parameters, an fs-laser can achieve high-quality preparation of microcavities on dentin surfaces. Zach and Cohen[30] found that a temperature rise higher than 5.5°C could cause irreversible necrosis of part of the pulp. The maximum temperature rise of the pulp and dentin surface was 0.6°C and 2.0°C during the whole processing in this experiment, which revealed good biosafety.

The SEM images indicated that in addition to several plow marks parallel to the siding direction, as observed in the Zheng et al.[21] study. A large area of exfoliation and cracks

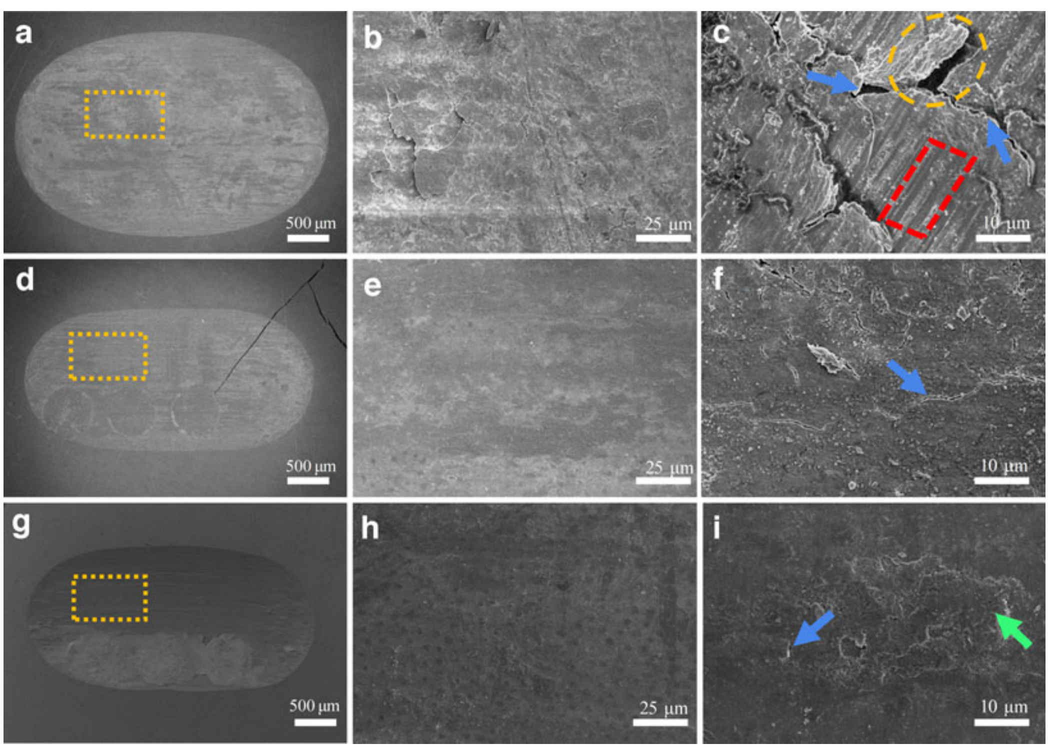


**FIG. 6.** Morphologies of the wear scars in the three friction pairs. From left to right, the three columns show the wear mode of dentin at magnifications of 30×, 600×, and 2000× in the three friction pairs. From top to bottom, three rows indicate blank dentin **(a–c)**, dentin in the wear scar of PICN group **(d–f)**, and dentin in the wear scar of amalgam group **(g–i)**, respectively.

Table 3. Paired T-Test of Wear Volume and Depth in Amalgam and Polymer Infiltrated Ceramic Network Groups

| | *Mean ± Standard deviation* | *95% Confidence interval* | t | *df* | p |
|---|---|---|---|---|---|
| Pair 1 | 164.17 ± 46.86 | 125.00–203.35 | 9.91 | 7 | <0.001 |
| Pair 2 | 137.00 ± 37.67 | 105.51–168.50 | 10.29 | 7 | <0.001 |
| Pair 3 | $(9.30 \pm 2.13) \times 10^8$ | $7.52 \times 10^8$–$11.08 \times 10^8$ | 12.37 | 7 | <0.001 |
| Pair 4 | $(8.39 \pm 2.60) \times 10^8$ | $6.22 \times 10^8$–$10.56 \times 10^8$ | 9.13 | 7 | <0.001 |

Pair 1, depth in amalgam group ($\mu$m); Pair 2, depth in PICN group ($\mu$m); Pair 3, volume in amalgam group ($\mu m^3$); Pair 4, volume in PICN group ($\mu m^3$).

was also observed in the blank dentin. The morphologies showed that the wear mode of dentin with titanium spheres was mainly abrasive wear and part of fatigue wear. After microfilling with PICN, the range and depth of the dentin partial exfoliation in the wear scar were obviously decreased. The fatigue wear of the dentin greatly weakened, and the strong plow effect could hardly be seen. In the amalgam group, abrasive wear and fatigue wear can solely be seen; instead, large cohesive particles appeared. A possible explanation might be that the introduction of another material changed the wear behavior of the dentin.

Since amalgam and PICN have a greater wear resistance than dentin, microscopically, a very shallow layer of the dentin is first ground off, and then, the filled materials become the prominent points that are ground against, with the antagonist in the next reciprocation cycle, thus reducing the stress distributed on the dentin and delaying its loss.[31] Different dentin samples have diverse surface properties, and so to obtain a relatively valid result, we included self-control for every sample and used a ratio to represent the improvement, which allowed us to eliminate individual differences.

Similarly, wear tests performed by Basnyat et al.[32] on a surface textured with a TiAlCN coating with molybdenum disulfide ($MoS_2$) showed a 50% increase in wear life. These authors also used microtexturing technology, while their substrate was steel. Although $MoS_2$ served as a solid lubricant in their study, new material was introduced to diffuse the stress, and we obtained a similar effect. Esteves-Oliveira et al.[12] also used a novel laser strategy to treat enamel and obtained good antierosion properties. However, they did not involve friction and wear tests, and we cannot imply that their wear resistance changed.

Tooth wear is a common phenomenon that exists throughout one's whole life. Once dentin is exposed, it wears away very quickly. Because the wear resistance of dentin is lower than that of enamel due to the lack of enamel rods and the reduction of hydroxyapatite content. There are no ideal treatments to decelerate its quick loss. We applied the microtexturing method to the surface of dentin and achieved great effect. Not only can the wear resistance be improved $3 \sim 4$ times, but the wear behavior of dentin was also changed totally. The depth of the microtexturing layer was only about 200 $\mu$m, far less than the preparation depth of a full crown. Meanwhile, fs-laser can realize "cold" processing on the tooth with negligible thermal degradation. So the preparation of microcavities was very minimally invasive compared with the traditional mechanical preparation. We believe it is an ideal minimally invasive approach to palliative treatment for severe dentin wear.

The present study has several limitations. First, for the microfilling materials, we used an amalgam and PICN.[33,34] Although the clinical application of amalgam has been gradually reduced due to aesthetic concerns, we still involved it in the experiment in light of its excellent wear resistance and plasticity to prove the feasibility of the new method that we explored. Composite resin and glass ionomer cement were not involved in this study because their wear resistance was not as good as dentin. Then, only the wear resistance of a certain proportion of microfilling of the dentin surface was measured, and the specific correspondence between the filling proportion and the increase in the wear resistance has not been involved. At present, the experiment is still in the early laboratory research stage.

In future clinical applications, an fs-laser device and a mechanical light-guiding arm will be needed to direct light from the laser device into the patient's oral cavity. Our research team has planned the method of clinical application in the future and designed it as an fs-laser oral therapy robot.[35–37] The specific fs-laser device limits its immediate usage in the dental office. This study is just the beginning of this research project and there remain a number of research topics.

## Conclusions

Overall, the wear resistance of the dentin was greatly improved with microcavities prepared by an fs-laser and filled with an amalgam or PICN. It can be inferred that after applying the strategy, the time before pulp exposure of a severely worn tooth can be doubled. Further study could assess the wear resistance improvement with antagonists of different materials to adapt to different oral conditions. The specific correspondence between the filling proportion and the increase in the wear resistance remains to be studied.

## Author Disclosure Statement

No competing financial interests exist.


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