

# Wear and damage at the bonded interface between tooth enamel and resin composite

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## ABSTRACT

**Objective:** To investigate the wear mechanisms and evolution of damage in tooth enamel-resin composite bonded interfaces caused by sliding contact, and to develop an understanding of interface degradation from a tribological viewpoint that supports clinical recommendations for improving interface integrity.

**Methods:** Reciprocating wear tests were performed on bonded interface samples involving commercial resin composite (Tetric N Ceram Bulk Fill), resin cement (Rely X U200) and tooth enamel using the ball-on-flat configuration. The bonded samples were subjected up to  $5 \times 10^4$  cycles of sliding contact, and the wear depth and wear track morphology were characterized after increments using white light interferometry and scanning electron microscopy, respectively. Optical microscopy was also used to evaluate cracks and their propagation in the samples.

**Results:** In the early stages of sliding contact, wear evolved most rapidly at the interface, followed by the enamel and the resin composite. Gradually, the difference between the wear depth at the interface and other areas decreased. Furthermore, cracks and brittle fracture appeared in the enamel during the early stages of wear, adjacent to the interface. With continuing cyclic loading, enamel wear manifested primarily as ploughs, with discontinuous pits and peeled material. Cracking decreased to only a few cracks extending to the inner enamel and parallel to the interface.

**Conclusions:** Cracking and damage occurred in the enamel during the early stages of sliding contact and accelerated by poor margin finishing. Cracks caused by wear under sliding contact could be one of the reasons for secondary caries and tooth discoloration.

## 1. Introduction

According to the modified United States public health service (USPHS) criteria, a successful resin composite restoration should have no cracks or fractures, ideal anatomical form, good color match, smooth surface texture, and also perfect marginal integrity [1]. The integrity of a restoration's margin is determined by the bonded interface, which consists of three parts, namely the tooth structure, adhesive, and the restoration. In clinical evaluations, the bonded interface is considered "intact" if there is no visible evidence of ditching along the margin, probing of the margin cannot detect a gap, and neither the tooth structure or the restoration have evidence of fracture [2,3]. A laboratory evaluation of this condition by scanning electron microscopy (SEM) indicates that the tooth structure, adhesive, and restoration are

tightly bonded, there are no gaps or hangers in the interface, and no cracks or fractures in the tooth structure and restorative material [4].

Due to the greater emphasis on minimally invasive restorative procedures and esthetics, resin composites have become the primary material for restorations in posterior teeth [5]. However, clinical trials have reported that composite restorations do not achieve the modified USPHS criteria, and largely due to degradation of the marginal integrity. For instance, clinical trials reported that the percentage of margins with a crevice caught by an explorer increased to 5% after 3 years [6]. The percentage of margins with defects increased to 11.5% after 4 years [7], and between 84%–92% after only 8 years of clinical use [8]. Collectively, these reports suggest that degradation of the margins is problematic, and that the breakdown of the bonded interface mainly occurs on the occlusal surface of Class I and Class II cavities

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[9–11].

Degradation of the bonded interface integrity can cascade into various problems, such as marginal discoloration, secondary caries, dentin sensitivity, pulp gingivitis, and even fracture of the restoration [12]. In fact, a decrease in the marginal integrity is one of the primary contributions to direct and indirect restoration failures. Carefully monitoring the bonded interface is one approach to extend their clinical longevity [13]. In recognition of the significance of this issue, *in vitro* studies have focused on marginal breakdown and bonded interface degradation. Some studies have reported that microleakage causes destruction of the bonding interface [14,15]. Polymerization shrinkage stress causes gap formation and subsequent marginal microleakage at the enamel-adhesive interface. This leads to the deterioration of the bond and accelerates failure [16]. However, different view has been put forward in recent years. Ferracane and Condon reported that cyclic loading causes wear of the composite and tooth enamel, which extends to breakdown of the margins [17]. Garcia reported that wear phenomena superpose with other aspects of margin quality, and that gaps between enamel and resin composite did not play a major role in the breakdown of margins [18]. Frankenberger found that restorations with perfect margins at baseline deteriorate in clinical use, and that the majority of restorations with imperfect margins have distinct wear tracks due to the occlusal contact. He proposed that wear is the main reasons for decreasing “restoration integrity” [19]. Therefore, the breakdown of bonded interfaces is a function of occlusal loading and wear.

*In vitro* testing of tooth-biomaterial interfaces can serve as appropriate preclinical screening [20] and there is a substantial body of literature focused on wear of the bonded interface. Most reported studies have discussed wear properties of the adhesive and the factors affecting breakdown of the margins, but they did not evaluate wear of the interface itself [21–26]. While a few studies have commented that wear contributes to breakdown of the bonded interface [27], an explanation of the wear mechanisms and the process of damage initiation and evolution has not been presented.

If indirect restorations are placed by a skilled clinician, then the bonded interfaces are initially intact with very high integrity of the margins. Thereafter, contact sliding and wear contributes to the transition from high integrity to a state of degradation that may sacrifice the restoration. This process appears to be poorly understood. Therefore, the primary objective of this investigation was to investigate the wear mechanisms and evolution of damage in bonded interfaces involving enamel and resin composite under sliding contact. The goal was to generate an understanding of interface degradation from a tribological viewpoint that supports clinical recommendations for maximizing interface integrity. The hypothesis of this study is that wear damage may be more serious in tooth enamel near the interface, resulting in interface destruction.

## 2. Materials and methods

### 2.1. Materials

Bonded interface specimens were prepared involving tooth enamel and resin composite. The process involved etching of the enamel using 35% phosphoric acid gel (Eco etch, Ivoclar Vivadent, Liechtenstein), followed by application of resin cement (Rely X U200, 3M, America) and bonding to a commercially available resin composite (Tetric N Ceram Bulk Fill, Ivoclar Vivadent, Liechtenstein).

### 2.2. Specimen preparation

The tooth enamel used for the bonded interface specimens and tooth enamel specimens were from human teeth. Both the collection and the experimental procedures involving the teeth were approved by the Research Ethics Committee of Sichuan University, China. All of the

teeth were collected with consent. Fifteen bonded-interface specimens, as well as five tooth enamel specimens, five resin composite specimens and five resin cement specimens were prepared according to the following procedures. Ten unerupted third molars, caries-free and enamel-intact, were collected from donors 18–25 years old, and preserved for less than three months in distilled water prior to sample preparation [28–31]. The crowns were separated from the roots at the cemento-enamel junction, and cut into sections along the mesial-distal axis using diamond abrasive slicing wheels and a slow-speed slicing machine (Struers Minitom; Struers, Copenhagen, Denmark) under continuous water coolant. A total of twenty tooth sections were prepared.

The resin composite (Tetric N Ceram Bulk Fill) was placed in pre-fabricated molds using 1 mm longitudinal increments and light-cured 1 mm away from one end, at the middle section of the specimen and the other end for 30 s with an LED-type light source (Bluephase, 800 mW/cm<sup>2</sup>, Ivoclar Vivadent), to form cuboid blocks 6 × 6 × 4 mm<sup>3</sup>. Twenty composite blocks were fabricated in total. Fifteen pairs of tooth sections and composite blocks were then selected randomly. The interior faces of the tooth and one of the 6 × 6 mm<sup>2</sup> composite surfaces were ground with #800 mesh silicon carbide abrasive papers (Struers, Copenhagen, Denmark) under continuous water irrigation to prepare similar rough surfaces. Thereafter, the abraded surface of the tooth enamel was etched with the 35% phosphoric acid gel for 20 s [32–34], rinsed for 10 s and air dried according to the manufacturer's recommendation. The resin cement was then mixed following the manufacturer's instructions and applied to the entire composite surface. Subsequently, the inner surface of the tooth section was positioned on the resin cement (Fig. 1), ensuring that the occlusal surface of tooth section was level with the face of the composite block. After removing excess adhesive, light curing was then performed for 40 s and 1 mm away from each side of the specimen with a calibrated unit (DP385C, 1000 mW/cm<sup>2</sup>, Hemaoinstrument inc, Foshan, China). To control the resin cement thickness, indicator papers with a thickness of approximately 10 μm were utilized (Fig. 1). A single indicator paper was positioned at the periphery of the bonding surface near the dentin side. After completion, the bonded specimens were stored in artificial saliva for 24 h at 37°C. Five resin cement specimens were fabricated by using the same molds as the resin composite specimens and light-cured for 40 s like the bonded interface specimens.

The fifteen bonded interface specimens, the five tooth sections, five composite blocks and five resin cement blocks were embedded in self-curing epoxy resins (Struers, Copenhagen, Denmark). For the bonded specimens, the interfaces were arranged to face outward. Similarly, the labial or lingual face of the tooth sections and the 6 × 6 mm<sup>2</sup> surface of the composite blocks were arranged to face outwards in their respective mounts. After solidification of the mounting material, the exposed surface of each specimen was ground with silicon carbide abrasive papers (Struers, Copenhagen, Denmark) in a decreasing order of abrasive size (#800, #1200, #2400, and #4000) under continuous water irrigation. Then, all of the specimens were polished sequentially using DiaDuo-2 (Struers, Copenhagen, Denmark) with 3 μm particle size for 5 min, followed by OP-S NonDry (Struers, Copenhagen, Denmark) with particle size of 0.04 μm for 5 min. The polishing was performed on a dedicated instrument (Struers, Copenhagen, Denmark) with felt cloths (Dac, Struers, Denmark) under constant lubrication, until achieving a mirror-like surface. Finally, all specimens were ultrasonically cleaned for 10 min in deionized water (KQ-50B, Shumei, Kunshan, China). All the bonded samples were examined using an optical microscope (BX51-M, Olympus, Japan) to measure the interface width at magnification of 20×, only specimens with the interface width less than 20 μm were selected for the wear tests.

### 2.3. Nanoindentation

The elastic modulus (E) and hardness (H) of the resin composite, resin cement and enamel was measured using a Tribolindenter (Hysitron

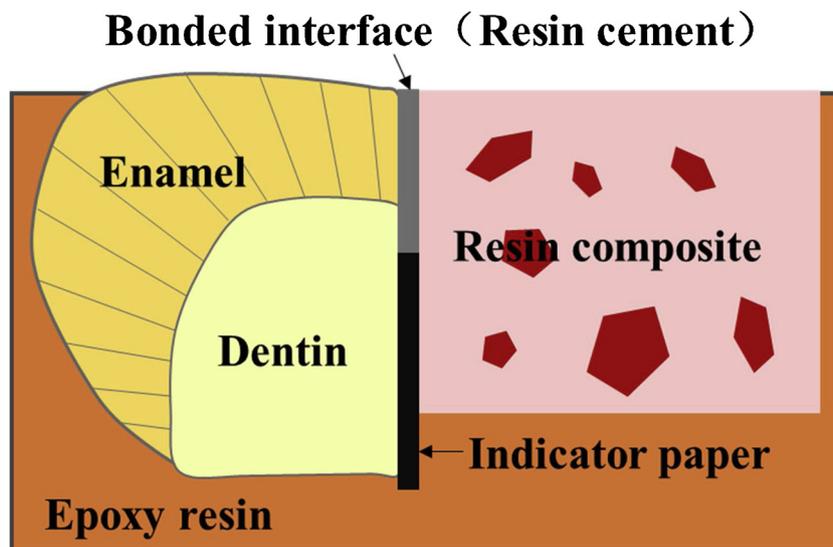


Fig. 1. Schematic description of the bonding process used for specimen development.

Triboscope, Minnesota, USA) with Berkovich diamond indenter having 100 nm nominal tip radius. All tests were performed at room temperature in the load-control mode and with maximum load of 2 mN. The loading was performed using a trapezoid function including a 5 s linear loading period, a 30 s hold period at the peak load, and a 5 s unloading period. Thirty indentations were made with an interval of 50  $\mu$ m between indentations on three of bonded interface specimens, tooth specimens, composite specimens and resin cement specimens, respectively. The values of E and H for each of the indentations were determined according to the Oliver and Pharr approach [35] and then averaged.

#### 2.4. Wear tests

Reciprocating wear tests were conducted using a commercial machine (MFT 5000, Rtec instruments Inc, USA), using a ball-on-flat contact mode. The experiments were performed at room temperature under simulated artificial saliva lubrication. The testing parameters included an imposed contact load of 20 N, sliding rate of 4 mm/s, articulation frequency of 1 Hz, and displacement amplitude of 2 mm. The displacement midpoint of the articulations was located at the bonded interface. Stylus alignment was ensured by the machine's function called "in situ". Following previous studies, ceramic balls of Si<sub>3</sub>N<sub>4</sub> were used as antagonists with a diameter of 6.35 mm [36,37]. For the bonded interface samples, a total of 5  $\times$  10<sup>4</sup> translation cycles were performed. In order to observe the evolution in wear response, three intervals of analysis were employed over the total loading history, including after 5  $\times$  10<sup>2</sup>, 5  $\times$  10<sup>3</sup>, and 5  $\times$  10<sup>4</sup> cycles [38]. Thus, five samples each were used to evaluate the wear characteristics after the three aforementioned periods of loading (3  $\times$  5 = 15 samples). After the loading was completed the wear tracks were scanned using a white light interferometer equipped with the tribometer, to calculate the wear depth (D) at locations of interest within the wear scar [39–41]. Three-dimensional topography maps of the wear tracks were reconstructed using the software that accompanies the profilometer (Gwyddion 2.30, Department of Nanometrology, Czech Metrology Institute). For the tooth, composite and resin cement specimens, the maximum wear depths of the wear tracks were recorded after 5  $\times$  10<sup>3</sup> translation cycles.

#### 2.5. Data analysis

One-way ANOVA was performed to assess the differences in E, H

and D between the enamel, resin composite and resin cement. Statistical analyses were performed using SPSS 18.0. A p-value of less than 0.05 was considered statistically significant.

#### 2.6. SEM observations

In order to analyze features concerning the progression of wear and the overall morphology of the bonded interface, a sequential analysis was performed over the 5  $\times$  10<sup>4</sup> cycles. Specifically, the bonded interface samples were ultrasonically cleaned and examined using a scanning electron microscope (SEM, INSPECTE, Czech, Republic) after 5  $\times$  10<sup>2</sup>, 5  $\times$  10<sup>3</sup>, and 5  $\times$  10<sup>4</sup> test cycles [42–45].

#### 2.7. Optical microscopy

After completion of the wear test, the samples were ground with silicon carbide abrasive papers (Struers, Copenhagen, Denmark) perpendicular to the bonded interface to reveal the subsurface region beneath the center of the wear scars. After reaching the appropriate location, the surfaces were polished sequentially using DiaDuo-2 (Struers, Copenhagen, Denmark) for 5 min and OP-S NonDry for 5 min as previously discussed under constant lubrication. Finally, the specimens were ultrasonically cleaned for 10 min in deionized water (KQ-50B, Shumei, Kunshan, China). Then all the samples were examined using an optical microscope (BX51-M, Olympus, Japan) to observe subsurface damage and cracks extending beneath the worn surface at the magnification of 20 $\times$  and 50 $\times$ .

### 3. Results

#### 3.1. Mechanical properties and wear depths of the three materials

Results for the elastic modulus, hardness and wear depth measurements of the three primary materials are presented in Table 1. The wear depths in this table were obtained at the location of maximum wear after 5  $\times$  10<sup>3</sup> cycles. The elastic modulus and hardness of the tooth enamel were significantly higher than those of the resin cement and composite, as expected. Although the mean elastic modulus and hardness of the resin cement was slightly lower than those of the resin composite, there was no significant difference. Nevertheless, the wear depths were different. The mean wear depths in the three materials were all significantly different, with decreasing wear depth from the resin cement, to the resin composite and finally to the enamel.

**Table 1**

Mean and standard deviation of the (E)lastic modulus, (H)ardness and wear (D)epth for the enamel, resin cement and resin composite. The wear depth is measured at  $5 \times 10^3$  cycles.

	E (GPa)	H (GPa)	D ( $\mu\text{m}$ )
The enamel	128.1 (7.7) <sup>a</sup>	8.5 (1.2) <sup>a</sup>	3.1 (0.5) <sup>a</sup>
Resin cement	15.6 (3.5) <sup>b</sup>	1.1 (0.2) <sup>b</sup>	10.2 (0.9) <sup>b</sup>
Resin composite	16.7 (1.1) <sup>b</sup>	1.1 (0.2) <sup>b</sup>	8.4 (0.5) <sup>c</sup>

Note: Data of the three materials marked as different letters in a column indicate significant differences ( $p < 0.05$ ).

### 3.2. Wear behavior

Three dimensional profiles of wear tracks caused by sliding contact with the ceramic ball antagonist are presented in Fig. 2. Three different instants of the wear history are presented in Fig. 2A through 2C including after  $5 \times 10^2$ ,  $5 \times 10^3$ , and  $5 \times 10^4$  cycles, respectively. All the wear tracks displayed the conventional oval-shape, deepest in the central region and shallow at the edges. The wear depth and wear volume increased with number of sliding contact cycles, as evident. However, the evolution of wear in each of the three materials were significantly different over the entire period of testing. At the early stage of sliding wear ( $5 \times 10^2$  cycles), the wear depth was greatest at the bonded interface, and the wear of the enamel and resin composite were lower, however wear of the enamel was much greater than that of the resin composite (Fig. 2A). With continuation of the sliding contact ( $5 \times 10^3$  cycles), the wear depths in the enamel, the interface and resin composite became more consistent (Fig. 2B). In the tertiary period of the loading history ( $5 \times 10^4$  cycles), wear of the composite was significantly greater than that of the tooth enamel and the wear track depth at the interface was between that of enamel and the composite (Fig. 2C).

A comparison of the wear track profiles at different intervals of sliding contact are shown in Fig. 3A, including at  $5 \times 10^2$ ,  $5 \times 10^3$ , and  $5 \times 10^4$  cycles. These profiles were obtained from near the center and along the major axis of the wear tracks. At  $5 \times 10^2$  and  $5 \times 10^3$  cycles, the depth of wear was accentuated at the interface and adjacent enamel, which is clearly apparent with respect to the composite. The wear in enamel decreased with increasing distance from the interface and wear of the resin composite, was substantially lower in comparison. However, at  $5 \times 10^4$  cycles, the wear track no longer shows an accentuated region of damage at the interface. At this more advanced stage, the composite exhibited the largest depth of wear, followed by the interface and then the enamel. Histograms comparing the wear depth measurements at the three intervals of cyclic loading are presented in Fig. 3B. The wear depth measurement for the resin cement

was selected at the location of the midpoint of the cement layer. For the tooth enamel and composite, the wear depth measurement was selected at a distance of approximately  $20 \mu\text{m}$  from the boundary of the interface. At  $5 \times 10^2$  and  $5 \times 10^3$  cycles there was no significant difference in the depth of wear between the three materials. But with evolution of the sliding contact cycles, the resin composite underwent significantly greater wear than the enamel and bonded interface.

### 3.3. Wear morphology

Micrographs detailing the morphology of typical wear tracks over the history of the wear process are shown in Fig. 4 through 6. Specifically, micrographs for the worn surfaces after  $5 \times 10^2$  cycles were shown in Fig. 4. Highlighted in this figure are the enamel, bonded interface and resin composite. Both the enamel and composite exhibited features of characteristic plough marks (Fig. 4B and C). Exfoliation of particles and abundant plough marks (arrows) were apparent on the surfaces of the enamel and composite, the latter oriented parallel to the direction of the antagonist movement. No cracks were evident at the surface of composite. A detailed view of the bonded interface is shown in Fig. 4D and to a great extent in Fig. 4E. The wear morphology of the resin cement and resin composite were very similar. These areas displayed an abrasive wear, preferential wear of the polymer matrix, and exposed fillers detached from the surface (Fig. 4D). However, characteristics of the adjacent tooth enamel were distinctly different from the resin composite in the wear track. Numerous cracks were apparent in the wear track, which extended to the interface, just like half of the parenthesis (black triangles in Fig. 4E). Degradation of the adjacent tooth enamel is clearly apparent at this higher magnification, and exhibits characteristics of brittle fracture. In comparison to farther from the interface, the width of the wear track in enamel near the interface was wider (Fig. 4D), suggesting a scuffing motion at the interface. Although the enamel-resin cement interface remains intact, the adjacent enamel has undergone fracture (Fig. 4D).

Micrographs detailing the morphology of typical wear tracks after  $5 \times 10^3$  cycles are shown in Fig. 5. With further evolution of the sliding contact, characteristics of the wear damage in the composite had no obvious change. Distinct qualities of abrasive wear were apparent in the composite, including numerous plough marks. However, damage of the enamel was different from that at  $5 \times 10^2$  cycles. The enamel was covered with an abundance of wear debris (Fig. 5B), which occupied nearly the entire surface, except for the region near the bonded interface as evident in Fig. 5C. Within this exposed region, it was possible to view microcracking in the enamel. In comparison to features evident after  $5 \times 10^2$  cycles, the quantity of cracks evident in the enamel decreased substantially, meanwhile the damage became concentrated just adjacent to the interface (black triangle in Fig. 5C). It appears that the

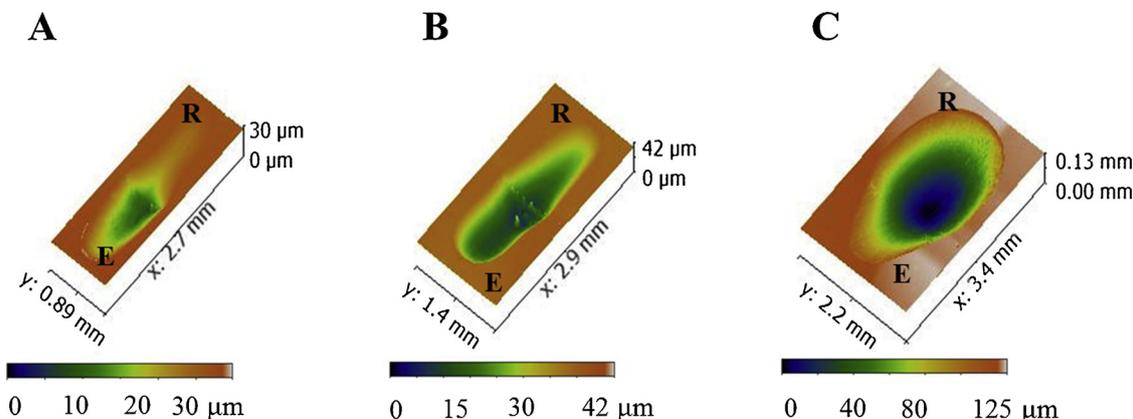
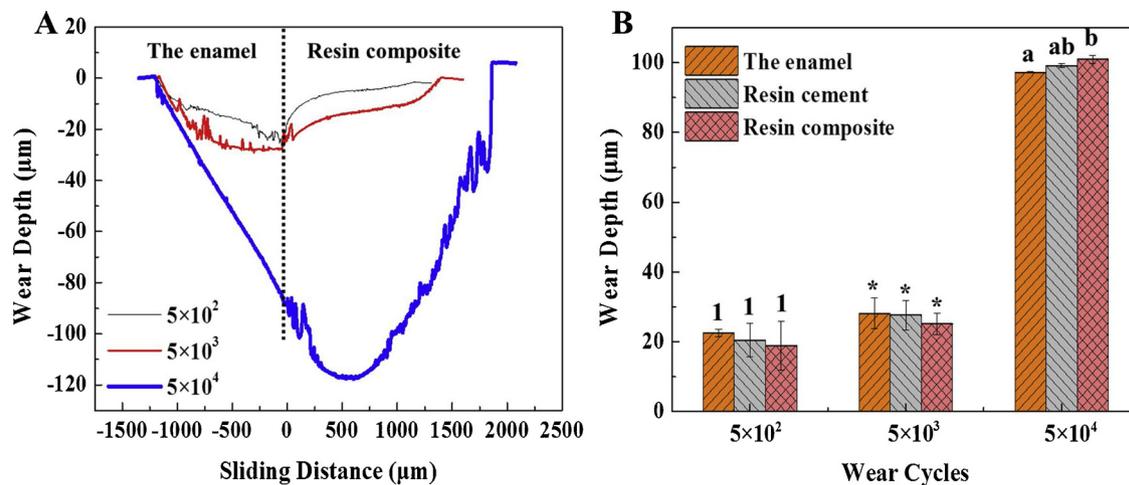
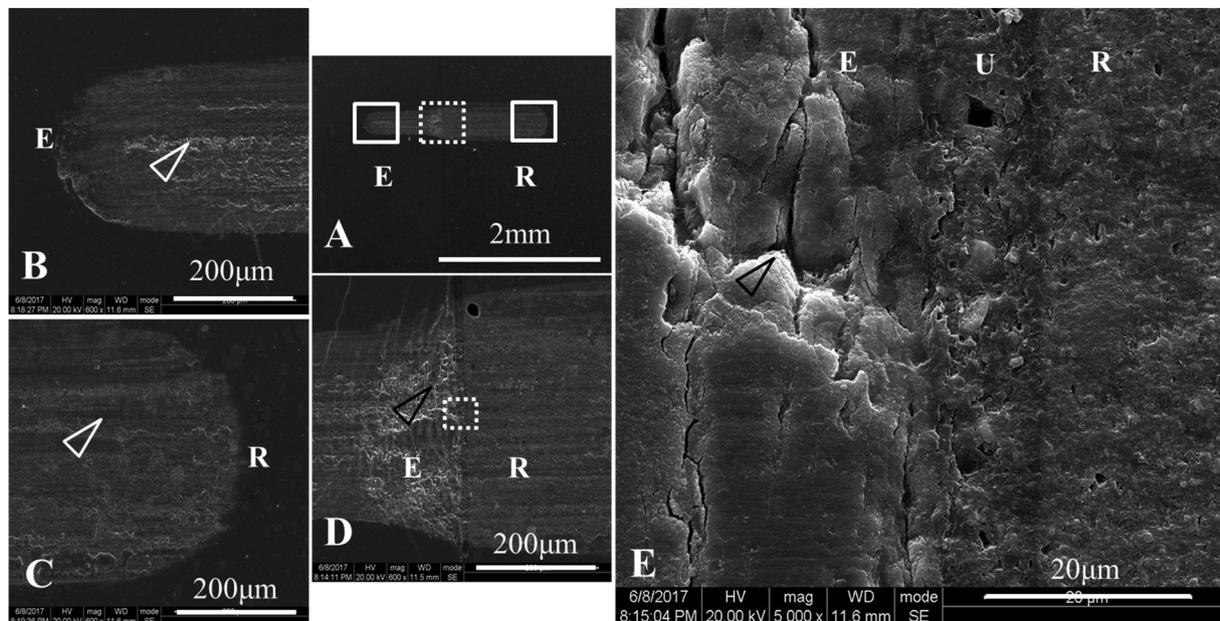


Fig. 2. Reconstructed three-dimensional maps of the wear tracks for specimens at A)  $5 \times 10^2$  cycles, B)  $5 \times 10^3$  cycles, and C)  $5 \times 10^4$  cycles, respectively. E: the enamel. R: resin composite. Note the distinct transition in depth of wear from the enamel to the bonded interface and resin composite in A and B.



**Fig. 3.** A comparison of depth profiles and wear depth histograms for different regions of the wear scar at different intervals of the cyclic wear history. A) Wear depth profiles at the three increments of evaluation. The dotted line indicates the bonded interface. B) Magnitude of the wear depth after three periods of articulation. Identical numbers, symbols or letters indicate no significant difference ( $p > 0.05$ ).



**Fig. 4.** SEM micrographs of a representative wear track after  $5 \times 10^2$  cycles. A) wear scar across the enamel and composite in the wear scar. White triangles indicate ploughs; black triangles indicate cracks. E: the enamel. U: resin cement. R: resin composite. The individual parts and the interface of the scar are highlighted and shown at higher magnification in (B), (C) and (D) respectively. The white dotted square in (D) outlines the interface area of the wear scar and is shown at higher magnification in (E).

integrity of adhesion between the tooth enamel and resin cement degraded. The enamel appears debonded and exfoliated from the wear surface, whereas the resin cement appears to be intact and exhibit higher integrity overall (Fig. 5D).

A representative wear track after  $5 \times 10^4$  cycles is presented in Fig. 6. Characteristics of wear in the enamel and composite were similar to those at  $5 \times 10^2$  cycles, with features indicative of abrasive wear, most notable from the shallow plough marks as shown in Fig. 6B and 6C (white triangles). At the bonded interface, the wear features within the resin cement and nearby composite appeared consistent with those from earlier observations in the wear process. The characteristics of damage in the enamel varied (Fig. 6B), but appeared more uniform and consistent with that of the marginal and middle areas of the enamel in the wear track. The enamel exhibited abrasive wear, with plough marks parallel to the direction of antagonist movement (white triangles in Fig. 6C). At the bonded interface, the extent of brittle fracture was

lower (Fig. 6D), and only a small degree of discontinuous pits and peelings were noted in the nearby tooth enamel (black triangle). At this depth of wear, the bonded interface between the resin cement and enamel appeared intact again (Fig. 6D).

### 3.4. Crack propagation

Profiles of the sectioned bonded interface samples after the three different intervals of cyclic sliding contact are presented in Fig. 7. In the early stage (Fig. 7A), no cracks were evident in the subsurface region of the composite or the tooth enamel. However with progression of the sliding wear, cracks were observed within the enamel and extended longitudinally parallel to the enamel rods adjacent to the bonded interface, (black triangle in Fig. 7B). With the increase in sliding wear cycles, the cracks extended more deeply (black triangle in Fig. 7C).

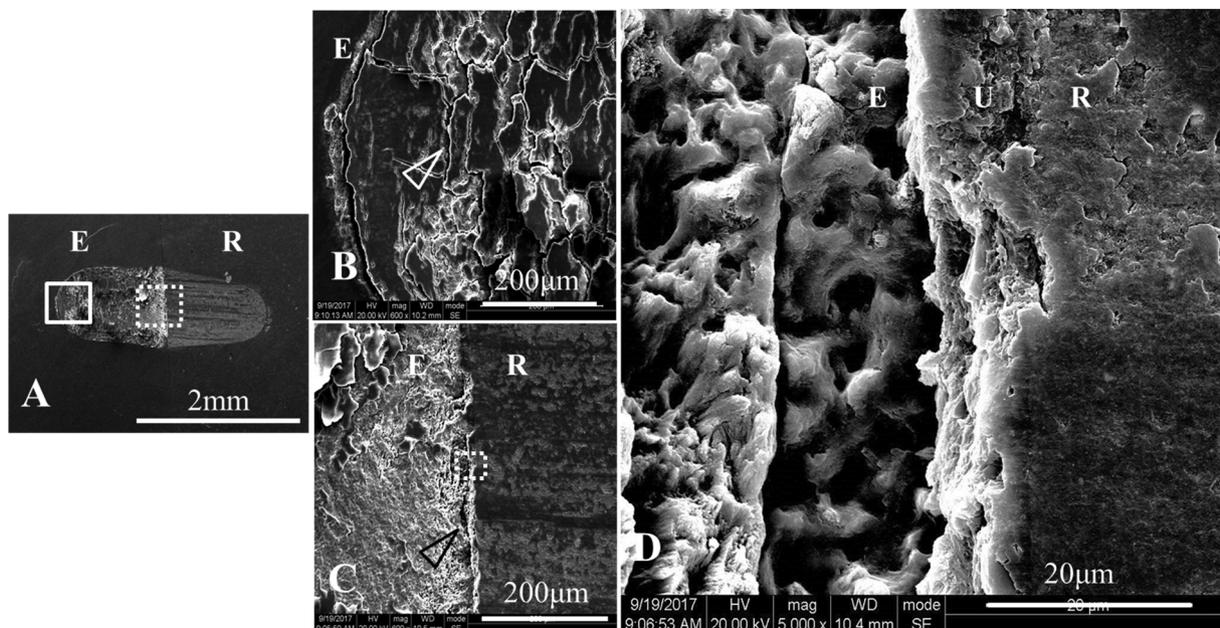


Fig. 5. SEM micrographs of a representative wear track after  $5 \times 10^3$  cycles. E: the enamel. U: resin cement. R: resin composite. A) Wear scar with highlights of the marginal area of the enamel in the wear scar (B) and the interface (C). White triangles indicate wear debris; black triangles indicate brittle fracture. The interface in C is shown at higher magnification in (D).

#### 4. Discussion

One of the most common criteria for assessing the clinical success of restorations is whether the bonded interface is intact. It is also one of the best measures for forecasting the long-term success [46]. Adhesive bonds between resin cement and tooth enamel can achieve a strength of 50 MPa or more [47], which meets the requirements for clinical use. However, degradation of the bonded interface is a remaining concern, especially at the occlusal surface, which is subjected to concentrated cyclic stresses [11]. Due to the articulating action of the occlusal forces, the bonded interface region at the margins experiences wear, and is considered a primary contribution to bonded interface failures [48].

Hence, understanding the wear behavior and failure mechanism of the bonding interface under sliding contact wear from a tribological viewpoint could help guide the development of clinical practices that extend the service life of bonded restorations.

At the occlusal surface, the bonded interface consists of three components including the enamel, adhesive and restoration. Matching the mechanical and tribological properties of the three materials, including the elastic modulus and hardness, is important to both the overall resistance to wear and the potential mechanisms that may contribute to its degradation. An ideal restoration should have mechanical and tribological properties similar to the dental tissue [38]. The elastic modulus and the hardness of the composite were

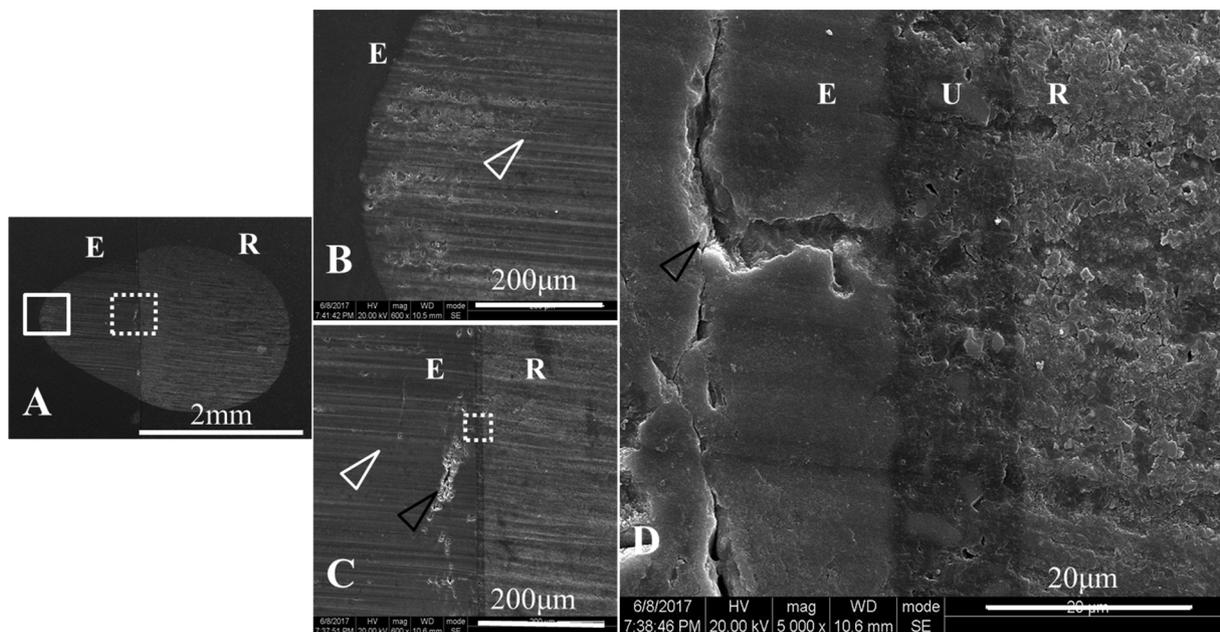


Fig. 6. SEM micrographs of a representative wear track after  $5 \times 10^4$  cycles. A) Wear scar with highlights of the marginal area of tooth enamel in the wear scar and the bonded interface, which are shown separately in (B) and (C). E: the enamel. U: resin cement. R: resin composite. White triangles indicate ploughs; black triangles indicate pit peeling. A higher magnification view of the interface is shown in (D).

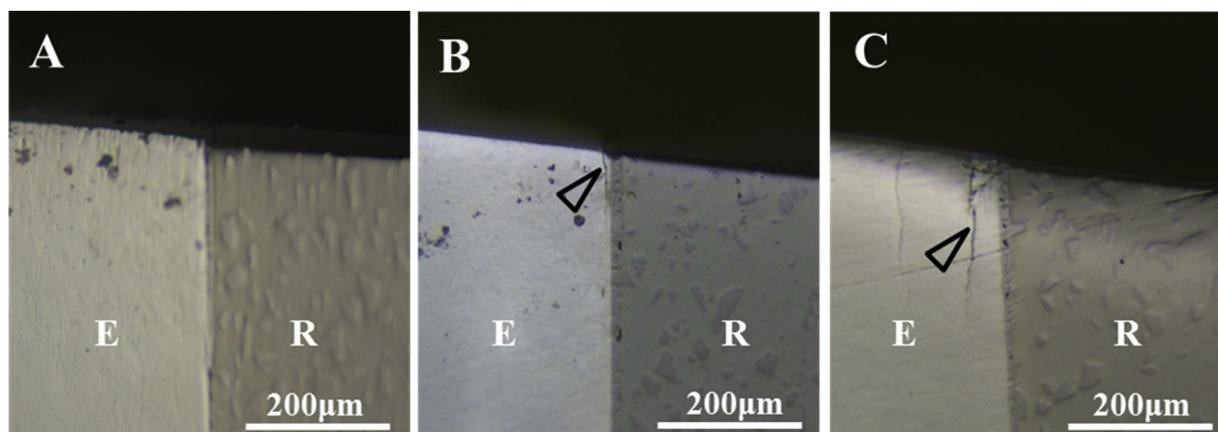


Fig. 7. Profile views of the bonded interface from optical microscopy at A)  $5 \times 10^2$  cycles, B)  $5 \times 10^3$  cycles, and C)  $5 \times 10^4$  cycles, respectively. E: the enamel. R: resin composite.

$16.6 \pm 1.1$  GPa and  $1.0 \pm 0.2$  GPa, respectively, consistent with those reported in previous studies [49]. In addition, the depth of wear in the resin composite after  $5 \times 10^3$  cycles was  $8.4 \pm 0.5$   $\mu\text{m}$ , which was in agreement with previous research [50]. Yet, the elastic modulus and hardness of the composite were substantially lower than those of tooth enamel ( $128 \pm 8$  GPa,  $8.5 \pm 1.2$  GPa). The composite also exhibited significantly lower resistance to wear than the enamel after the “steady-state” response was established. A mismatch of the moduli between the composite and tooth enamel causes a contact stress concentration within the enamel and adjacent to the bonded interface [51], which impacts the friction and wear behaviors. There are a variety of restorative materials available today such as resin composites, ceramics and polymer infiltrated ceramic network materials, each with their specific mechanical and tribological properties. A clinician must select the most suitable restorative material, which includes matching the mechanical and tribological properties of the tissue and restoration. Large occlusal contact forces necessitate a restoration with higher elastic modulus and hardness, more wear resistance.

Equally relevant, the mechanical properties of the adhesive contribute to the bonded interface durability. The bonded interface acts as a buffer to reduce the stress concentration between the tooth structure and restoration due to the mechanical property mismatch or the stress generated by resin shrinkage. A resin adhesive with lower elastic modulus and hardness has greater potential for stress relief [52]. However, as the elastic modulus and hardness decrease, the residual stress of the adhesive caused by polymerization shrinkage will also increase, which could accelerate the adhesive failure [53]. This suggested that the clinician should select the adhesive of appropriate mechanical properties according to different conditions.

Analyzing the bonded interfaces via SEM after  $5 \times 10^2$  cycles and  $5 \times 10^3$  cycles showed that there are two primary damage modes in the enamel, including abrasive wear and brittle fracture. The latter form of damage is localized within a few hundred micrometers of the bonded interface. Only abrasive wear was noted in the resin composite (Fig. 4 and 5). The disparity in damage between the two materials must result from the response difference in contact stress, i.e. associated with the differences in elastic modulus and hardness, as well as the fracture toughness. The latter is of substantial importance to the enamel and the development of cracks.

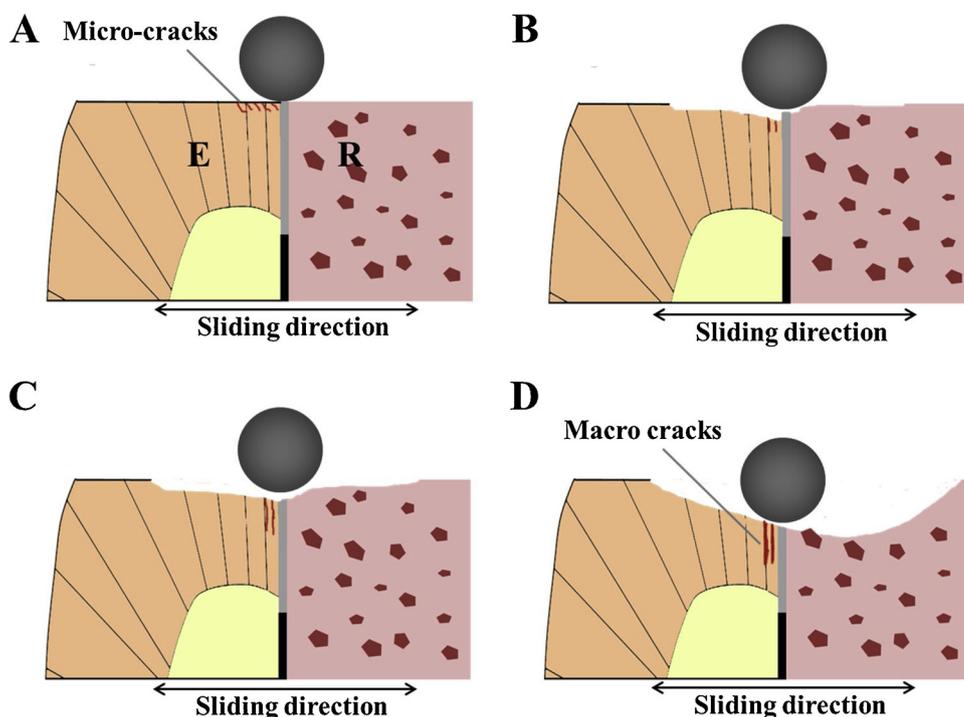
Tooth enamel consists of approximately 95 vol % mineral, with the remainder consisting of organic proteins and water [54]. The principal microstructural feature of enamel is the rods, which are approximately 5 to 8  $\mu\text{m}$  in diameter and consist of an assembly of needle-like apatite crystals that are consolidated into rod shapes by a very thin layer of enamel proteins. Near the surface of the tooth the rods extend towards the DEJ as a series of parallel columns, akin to a dense forest of bamboo. However, in the inner enamel the rods are “decussated” into a

pattern that has received attention due to its capacity for bestowing crack growth resistance. Near the occlusal surface and within the outer enamel cracks extend preferentially along the interface of adjacent rods due to the limited number of toughening mechanisms available for resisting crack growth [55]. Cracks that initiated under contact propagated along the enamel rod interfaces, which was evident from the occlusal views of the wear scars (Fig. 4D, 5D). Indeed, the cracks initiate and propagate in tooth enamel more easily, which was evident in the enamel near the interface (Fig. 7). While the fracture toughness can reach over  $2.5 \text{ MPa}\cdot\text{m}^{1/2}$  within the inner enamel of human teeth, it is less than  $1 \text{ MPa}\cdot\text{m}^{1/2}$  near the occlusal surface. In measures involving use of the indentation fracture resistance the fracture toughness of enamel ranges from roughly  $0.44\text{--}1.55 \text{ MPa}\cdot\text{m}^{1/2}$  [56], which is approximately half that of most resin composites. Thus, the comparatively low fracture toughness of the outer enamel makes it more susceptible to contact cracking under the concentrated shear stresses induced by sliding contact across the interface. Assuming that the coefficient of friction cannot be changed, the most viable means to reduce the shear stress at the interface is to increase the elastic modulus of the resin composite.

Under sliding contact with the antagonist ceramic, the polymer matrix of the resin composite undergoes deformation and wear preferentially, which exposes the fillers and enables their detachment [50]. The fracture toughness of the composite ( $2.4\text{--}3 \text{ MPa}\cdot\text{m}^{1/2}$ ) is effective at resisting the development of cracks under the concentrated shear forces initiated by contact and sliding [57,58]. Even under the highly-concentrated stresses at the bonded interface, the wear characteristics of the composite are similar to that of the resin cement.

The wear damage mode of the enamel evolved with progression of cyclic contact. As described previously, the mismatch in mechanical and tribological properties between the composite and tooth enamel resulted in a stress concentration at the interface. Within the early stage of the experiment ( $N \leq 5 \times 10^3$  cycles), the ball-on-flat point contact between the antagonist and specimen resulted in a small contact area and a high contact stress [59]. Under this high stress condition, cracks developed along the interface of the enamel rods on the wear surface and extended beneath the surface; in some instances the cracks caused brittle fracture (Fig. 4C and 5C). With progression of the test the contact conditions changed from point contact to concentric distributed contact. That resulted in a decrease in stress [60] and a reduction in cracks and brittle fracture at the interface. In the final stages of the sliding contact, plough marks developed in the enamel as shown in Fig. 6C.

Based on the experimental results, the evolution of damage at the bonded interface can be summarized according to the schematic description in Fig. 8. The experiment started as two-body wear. They potentially evolved to three body wear with the development of wear debris. At the early stage of sliding contact (Fig. 8A), concentrated



**Fig. 8.** Schematic representation of the wear and the evolution of damage at the enamel-resin composite bonded interface. E: the enamel. R: resin composite. A) The process begins with the development of short microcracks within the enamel and a shallow wear track. B) The process continues with more progressive wear in the enamel and the evolution of the microcracking. C) Wear progresses to develop within the enamel and within the resin composite, and microcracking depths in the enamel continue to extend farther sub-surface. D) Cracking in the enamel evolves to macro-cracking into the enamel and wear of the resin composite accelerates and begins to exceed that in the enamel.

stresses at the interface result in numerous cracks in the enamel. These cracks are rather superficial and concentrated beneath the surface of the wear scar. The cracks undergo extension, and small bundles of enamel rods undergo crushing, which leads to brittle fracture (Fig. 8B). Without support of the tooth enamel, wear within the resin cement and the resin composite undergoes an acceleration (Fig. 8C). The depth of wear in the enamel exceeds that of the composite due to this concentrated stress, whereas the depth of wear at the interface is much higher than those in other areas of the wear track (Fig. 3). However, as the wear process continues, the contact stress in the enamel decreases due to brittle fracture, the increase in compliance caused by the interfacial cracks and the development of a concentric contact geometry. That transitions wear to the interface and the resin composite. With progression of wear, the dominant cracks in enamel remain near the interface and undergo further propagation as a result of the cyclic stresses. Previous research has reported that tooth enamel is more wear resistant than resin composite under the same experimental conditions [61]. Indeed, that is true within the latter period of wear, which is evident when the depth of wear in the enamel is lower than that of the composite (Fig. 3A and B).

After placement of a restoration, the clinician often needs to adjust the occlusion. An appropriate occlusion requires that no premature contact or occlusal interference exists on the restoration when the patient bites. That process is essential to prevent problems such as fracture of the restoration or tooth due to excessive stress, increased tooth mobility, abnormal muscle function, and even temporomandibular joint disorders [62–64]. Results of this study showed that the stresses that develop at the initiation of sliding contact between the ceramic ball and the bonded interface were large enough to cause brittle fracture within the enamel adjacent to the interface, even in perfectly adjusted occlusion. The consequent damage had a substantial influence on the friction and wear behavior of all three components of the interface at the early stages of sliding contact, which led to an acceleration of wear overall. Clearly the clinician should not only prevent premature contact at points and by occlusal interference, but also be vigilant to avoid point contact between the bonded interface and opposing tooth pair. A distributed area contact is recommended to clinicians when adjusting the occlusion in regions where high occlusal forces are expected.

The traditional view is that the microleakage at the margins of resin composites is mainly caused by polymerization shrinkage, as well as the different thermal expansion coefficients between the resin composite and tooth structure. Contraction caused by cold temperatures leads to tensile forces between the hard tissue foundation and the restoration, which could lead to the formation of gaps and microleakage [65]. Furthermore, cyclic sliding contact can foster hydraulic pumping, bringing fresh media and bacteria into the gap, and possibly removing some of the saturated solution and bacterial waste from the gap, which aids bacterial penetration [66]. In this study, cracks formed in enamel during the early stage of cyclic contact and gradually extended with an accumulation in cycles. These cracks form small gaps under cyclic loading, leaving the interface more susceptible to biofilm degradation by helping bacteria access the dental tissue, and potentially resulting in secondary caries and tooth discoloration. If cracks extend more deeply they can cause postoperative sensitivity and even pulpitis [67]. Hence, the results provide an additional contributing mechanism for the formation of gaps and microleakage, which lead to interface destruction.

Although care was taken to study the mechanisms of wear and damage at the bonded interface in a clinically relevant manner, it is important to recognize the differences between the test conditions and the clinic. For instance, the bonded interfaces in the experiments were prepared under very controlled conditions, with specific geometry. The bonded interface thickness (i.e. the thickness of the resin cement layer) in clinic varies [22], and the type of adhesive used as well. Different adhesive thickness and types could contribute to the stress distribution at the interface and bond strength in a number of ways [68,69]. Whether the interface width and the type of adhesive will affect the wear resistance of the bonded interface is relatively unknown. In addition, the wear process was conducted under a neutral pH condition, which does not reflect the oral environment of all patients. Variations in pH and acidic conditions should be considered. As such, follow-up experiments are currently underway that consider the influence of these factors on the wear behavior of the bonded interface.

## 5. Conclusions

An experimental evaluation of the wear mechanisms and evolution

of damage in bonded interfaces of enamel and resin composite caused by sliding contact were analyzed. Within the limitations of this study, the following conclusions were drawn:

- 1 Within the early period of sliding contact (first  $5 \times 10^2$  cycles), wear was significantly greater at the interface than in the enamel or resin composite. However, with further progression of the cyclic sliding contact, the difference in wear depth between the bonded interface and other areas decreased.
- 2 With progression of sliding contact ( $5 \times 10^3$  cycles), the wear depth became consistent in the three materials, after which the wear progressed more rapidly in the resin composite.
- 3 The wear damage within the vicinity of the interface was unique from the other regions of the wear track and was most extensive in the enamel. Numerous cracks and characteristics of brittle fracture were identified, with cracks extending a few hundreds of micrometers in depth, parallel to the bonded interface. As wear of the enamel progressed, the damage manifested as plough tracks and was accompanied with discontinuous pits and peeling.

### Declarations of interest

None.

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