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In situ observation on the failure behavior of ZrO$_2$-resin-dentin bonding interface with prefabricated indentation defects

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Abstract

Interfacial cracking and fracture of restorative materials are major obstacles to realize effective dental restoration. Especially, the bonding failure of compound interfaces consisting of dentin, resin and zirconium dioxide (ZrO$_2$) ceramic, exhibit complexity, variability and unpredictability due to the complicated loading type and oral environment. By using a self-developed miniaturized horizontal device integrating with thermostatic artificial saliva, an approximate oral environment was established to investigate the failure mechanism of ZrO$_2$-resin-dentin compound interfaces. Through real-time in situ observation of shearing deformation behaviors of the dentin-resin and resin-ZrO$_2$ interfaces, the continuous propagation processes of cracks nucleating from the interfacial intersection line or dentin’s interior were analyzed in detail. The discontinuous cracking behaviors revealed the prior crack of resin-ZrO$_2$ interface, which attributed to the acid etching of dentin and significant gradients in Young’s modulus and hardness compared with the corresponding parameters of dentin-resin interface. The significant interfacial differences in mechanical properties promoted the crack nucleation and induced the bonding failure. A widest crack with a width of 1.4 μm inside the dentin was also observed from the fractured ZrO$_2$-resin-dentin specimen. This paper focused on the discontinuous interfacial cracking behaviors and bonding failure mechanisms of ZrO$_2$-resin-dentin specimen, which would be beneficial to the research of novel composite resins and the improvement of bonding processes.

1. Introduction

Dental bonding restoration is an effective therapeutic method to directly cure tooth fractures, dental caries and tooth erosion. Although a series of dental restorative materials, including metals [1] (Ni–Cr Alloy, Co–Cr Alloy and Ti Alloy, etc), composite resins [2–4] and ceramic materials [5, 6] (feldspar ceramics, Al$_2$O$_3$ ceramics and ZrO$_2$ ceramic, etc), have been widely used, the strength and reliability of bonding interface need to be further improved. Regarding the dentin restoration, although the demineralization process by acid etching [7] and wettability enhancement could promote the infiltration of resin and produce cured mixed layer with thickness with range from 2 μm to 10 μm [8], the interfacial cracking is still a major problem far away from mature solution. The continuous interfacial cracking behavior and critical failure mechanism need to be taken into account. Meanwhile, the dentin restoration in clinical scenario based on ZrO$_2$ ceramic and resin exhibited advantages in strength, manufacturability, appearance, wear resistance [9] and thermal stability [6], due to the stable chemical properties, high strength, excellent crack resistance and biocompatibility of ZrO$_2$ ceramics. The infiltration effect of resin into the microporous structure of dentin could also realize the enhancement of bonding strength [10].
Considering the variability of oral environment, service temperature, multi-oriented and static-dynamic coupling characteristics of chewing forces [11, 12], the bonding cracking failure of ZrO₂-resin-dentin compound interfaces exhibited the diversity of failure behaviors [13, 14]. Meanwhile, as a typical brittle material, ZrO₂ ceramic might suffer from fracture risk due to the internal defects and variable loading types, including hard particle scratch, interfacial shearing, surface friction and compressive cyclic loading [15]. The observation of crack nucleation and propagation behaviors of compound interfaces was particularly important for understanding the failure mechanisms [9, 16]. Especially, the real-time observation of crack evolution [17–19] could reveal the discontinuous behaviors of compound interfaces and confirm the critical stress or strain conditions leading to the nucleation of microcracks, which was beneficial to the researches of novel composite resins development and the improvement of bonding processes [20]. Regarding the testing of bonding behavior and failure mechanism of interfaces, etching steps and mechanical loads were important factors determining the bonding strength [21]. Frankenberger et al systematically investigated the marginal integrity of dentin adhesives subjected to thermo-mechanical loading, through the transmission electron microscopy analysis of a large number of fracture morphologies, a two-step self-etch adhesives was proved as optimal adaptation to dentin [22]. Luong et al revealed that low-viscosity resin improved the bonding strength of dentin, their tensile tests also verified that the bonding strength of dentin and resin composite significantly decreased after 10⁴ thermocycles [23]. Meanwhile, considering the actual service condition of compound interfaces, in situ testing could equivalently investigate the continuous interfacial shearing behaviors and bonding failure mechanisms [17, 24]. Furthermore, according to our previous studies, prefabricated defects with large stress concentration coefficient could be benefit to confirm the crack nucleation and facilitate the real-time observation of crack propagation [25].

Regarding the ZrO₂-resin-dentin compound interfaces, effective experimental methods are needed to directly observe the continuous micro deformation behaviors and reveal the bonding failure mechanisms. In this study, through the establishment of an experimental system integrating with approximate oral environment, miniaturized in situ testing device and constant temperature control unit, the real-time observations of evolution processes of the dentin-resin interface and resin-ZrO₂ interface, the crack nucleation and propagation behaviors and interfacial bonding failure mechanisms were experimentally investigated in detail. The interfacial difference in mechanical properties was considered as the main influencing factor to induce the bonding failure. The discontinuous fracture behavior of compound interfaces attributed to the interface differences and prefabrication of indentation defects was also regarded as a research hypothesis.

2. Materials and methods

2.1. Specimen preparation

Taking the crown section of premolars from 18 to 60 years-old adults as experimental materials, which were stored in deionized water at 4 °C [26] and subsequently immersed in normal saline solution containing 900 mg l⁻¹ NaCl buffered with 57.5 mg l⁻¹ CaCl₂. The immersion treatment in deionized water would prevent dehydration during the reservation process. Meanwhile, compared with artificial saliva, the immersion treatment in normal saline would prevent subsequent potential spoilage and performance degradation. As stated by Gustafson et al, the immersion in normal saline for a long time (6 days) will weaken the Young's modulus [27], therefore, the immersion lasted for 2 h to weaken the influences and maintain the equilibrium of Ca ion. Symmetrical double L-shape specimens were prepared as shown in figure 1. Ethical approval for the collection of premolars were obtained from institutional ethics review boards. Regarding the preparation procedures, clinically collected dentin with L-shape was adhered with ZrO₂ ceramic after etching by using light cured composite resin (3M-resin-250, mainly consisted of silanized zirconia, ethylene dimethacrylate, silica and 2-Hydroxy-4-methoxybenzophenone, producer: 3 M ESPE, Germany) and bonding agent (mainly consisted of 2-methyl-2-acrylic acid 1, 2-hydroxyethyl methacrylic acid, photoinitiator, 3 M AdperTM, producer: 3 M ESPE, Germany). Each ZrO₂-resin-dentin adhesive specimen was prepared through bonding a couple of L-shape dentin and ZrO₂ ceramic. Figure 1(a) illustrates the sampling direction of clinically collected dentin, which was cut from the direction perpendicular to occlusal surface. The enamel layer was removed through cutting and subsequent mechanical polishing. Precise saw cut (AUTOFOR, YC22) with thickness of 0.2 mm and external diameter of 22 mm was used to prepare rectangular dentin specimen. The rotation speed was set as 1000 r min⁻¹. Then, with the aid of two degrees of freedom precision positioning platform (Supereyes, Z007) integrated with digital microscope, the clinically collected extracted dentin (supplied by Dental Center, 964 Hospital of PLA) was firstly sliced into stepped L-shape with height difference of 1.5 mm, the inner side of the specimen's thinner part was the adhesive surface, which was cleaned ultrasonically for 10 s [28] and polished by using diamond abrasive pastes with decreasing particle sizes ranging from 5 μm to 0.5 μm. By using an optical microscope (Olympus DSX500) with a high depth of field and three-dimensional imaging function, the surface
profile of polished dentin side was captured as shown in figure 1(b), and an average surface roughness was measured as approximate 0.96 ± 0.24 μm through line profile analysis, the mean value of roughness was calculated based on 10 polished ZrO2-resin-dentin specimens and 50 randomly selected paths. Meanwhile, ZrO2 ceramic with the same L-shape was fabricated by following a sequence of steps involving design of L-shape 3D model, stereolithography and high temperature sintering. The adhesive surface of dentin specimen was acid etched before applying bonding agent by using phosphoric acid with concentration of 37% ± 2% (Gluma Comfort Bond, producer: Heraeus Kulzer, Germany), the acid-etch time lasted for 30 s, then dentin specimen was rinsed by distilled water. Then the bonding agent was brushed on the inner side of previously prepared L-shape dentin twice using with uniform brushing time of 15 s and interval time of 20 s followed by 20 s light curing. Then the resin was uniformly coated on both bonding surfaces followed by ultraviolet light curing lasting for 30 s [29]. As a matter of fact, as the ZrO2-resin-dentin specimens were placed on the 2D precision positioning platform, the tip of the light-cure lamp was right above the specimen’s surface with a constant height distance of 10 mm. With the aid of linear motion function of the platform, the relative motion trajectory of ZrO2-resin-dentin specimens was along the direction of the composite interface. The thickness of resin between dentin and ZrO2 was controlled by applying equivalent chewing force. A pre-compressive load of 100 N lasting for 5 min was directly applied on the compound interface through a compressive device integrated with S-type load sensor (BBS, Transcell), considering the natural chewing force with range from 30 N to 200 N [30]. As shown in figure 1(b), the average interface thickness was measured as 316.2 ± 36.7 μm.

In order to confirm the crack nucleation location and facilitate the observation of crack propagation, a series of prefabricated Vickers indentation defects were prepared on the dentin-resin interface (defined as Interface I) and resin-ZrO2 interface (Interface II), respectively. The prefabrication of Vickers indentation complied with the requirement of International standard: ISO 6507-1-2018 [31]. The prefabricated indentation defects would contribute to the analysis of the differential crack evolution behaviors at both interfaces. Based on identical indentation loads of 10 N and dwell time of 15 s, the residual indentation imprints exhibited different morphologies due to the differences in hardness and Young’s modulus of dentin and ZrO2 ceramic. Specifically, the residual morphology on Interface I manifested as a regular rectangle imprint. However, compared with Interface I, the differences in mechanical properties between ZrO2 ceramic and resin significantly affect the indentation morphology on Interface II, and the overall dimension of residual indentation was relatively smaller. Obvious plastic deformation accumulation was observed on the resin’s side. Meanwhile, the relatively smaller and sharper morphology on the ZrO2 ceramic’s side was attributed to the continuous sliding behavior of the Vickers indenter on the ZrO2 ceramic’s surface during the quasi-static indentation process.

2.2. Mechanical properties
Since the difference in mechanical properties affected the residual indentation morphologies, the critical condition leading to the crack nucleation could be definitely related to the surface mechanical properties. By using nanoindentation techniques (Agilent 200), a set of continuous testing locations along with a virtual path...
passing through and perpendicular to the two parallel adhesive interfaces were selected to obtain the Young’s modulus and hardness of various locations. Seen from figure 2(a), on basis of constant indentation load of 200 mN and dwell time of 20 s, significant differences in indentation responses were obtained, as the indentation load-depth curves exhibited different maximum depths $h_m$ and residual depths $h_f$ dependent with the testing locations. Regarding the ZrO$_2$ and dentin sides, the average $h_m$ obtained from the curves were calculated as 0.83 $\mu$m and 1.85 $\mu$m, respectively. The indentation responses of resin side exhibited an average $h_m$ of 5.64 $\mu$m.

Considering that the slope of unloading curve could determine the hardness and Young’s modulus [32], the gradient effect adjacent to the dentin-resin interface and resin-ZrO$_2$ interface might cause asynchrony of interfacial cracking behaviors. Specifically, 20 sets of indentation experiments were carried out in order to reduce the experimental error. As shown in figure 2(b), with regard to the ZrO$_2$, resin and dentin sides, the calculated Young’s modulus and hardness were 190.66 $\pm$ 23.62 GPa, 9.71 $\pm$ 2.12 GPa, 86.55 $\pm$ 13.28 GPa and 10.05 $\pm$ 2.2 GPa, 0.29 $\pm$ 0.27 GPa and 4.07 $\pm$ 1.12 GPa, respectively. The average modulus ratios of ZrO$_2$ to resin and dentin to resin were 19.63 and 8.91, respectively. The corresponding hardness ratios were 34.66 and 14.03, respectively. The significant gradients in Young’s modulus and hardness of resin-ZrO$_2$ interface might promote the crack nucleation and affect the bonding cracking failure [33].

2.3. In situ testing device
A modified miniature horizontal device integrating with tensile/compressive loading function [34] was used to carry out the equivalent shearing tests of ZrO$_2$-resin-dentin specimens. According to our previous study [35], a water bath integrated with a couple of heating rods and thermocouple was mounted at the specimen clamping region between a couple of symmetrical columns with flat steps. Seen from figure 3, a medium container filled with artificial saliva was placed inside the water bath. The thermostatic medium environment was achieved based on closed loop control of water temperature. Therefore, through integrating with the container filled with thermostatic artificial saliva, combined with the real-time observation of surface microstructure evolution by

![Figure 2](image-url)
using an optical microscope (Olympus DSX500) with a high depth of field and three-dimensional imaging function, an in situ testing system under an approximately thermostatic oral environment was established. When subjected to a quasi-static compressive load, considering the orientations of compound interfaces were parallel to the loading direction, the symmetrical double L-shape specimens sustained equivalent interface shear stress. During the tests, constant temperature of 36.8 °C and quasi-static strain rate of $10^{-4}$/s were set. The prepared double L-shape ZrO$_2$-resin-dentin specimen was clamped between a couple of support columns, and a pair of symmetrical flat steps on the columns were fabricated to weaken the misalignment during uniaxial testing. On basis of a driving unit consisting of a high-power servo motor, two-stage worm gears and a ball screw with left- and right-hand thread at the left and right ends, respectively, a maximum load of 2335 N, a uniaxial stroke of 8 mm and minimum loading speed of 10 nm s$^{-1}$ were achieved. During the imaging process, due to the self-locking function of worm gears, the imaging with high resolution could be obtained as the device could momentarily stop before imaging [36]. Meanwhile, the left- and right-hand thread of ball screw could also guarantee the relative stationary of indentation defects (as the observation target) during the shearing tests and imaging process.

3. Results and discussions

3.1. Discontinuous deformation behaviors

By using the established in situ testing system under approximate oral environment, the crack evolution behaviors nearby the prefabricated Vickers indentations defects on dentin-resin interface (Interface I) and resin-ZrO$_2$ interface (Interface II) were obtained through the real-time observation. When subjected to an equivalent interfacial shearing load, the compound interfaces exhibited different crack propagation and bonding failures behaviors [37]. As shown in figure 4(a), the load response showed segmented linear monotonic increasing trends. From initial loading to 160.8 N, the ZrO$_2$-resin-dentin specimen elongated linearly with a relatively large slope. From 160.8 N to ultimate fracture, the equivalent shearing load gradually increased with a relatively lower slope as a function of time, which indicated a discontinuous fracture process of the compound interfaces [38]. Combined with the optical morphologies of a couple of a group of parallel compound interfaces, the indentation microregions exhibited diversified deformation behaviors as illustrated in figure 4(b). On basis of external load of 93.9 N, regarding Interface I, plenty of multi-oriented microcracks nucleating inside the indentation imprint and a group of microcracks converging towards the Vickers indentation edge perpendicular to Interface I were observed on the sides of dentin and resin, respectively. The orientation difference was probably mainly attributed to the porous structure of dentin after acid etching treatment and isotropy and amorphous properties of resin. The porous structure of dentin effectively restrained the crack propagation due to the anti-cracking effect, and the stress concentration effect adjacent to the indentation edge at resin side facilitated the convergence of microcracks. Meanwhile, the microcracks nucleation adjacent to the interface exhibited approximately parallel orientations attributed to the significant differences in interface mechanical properties. The main crack continuously propagated until a significant main crack formed along Interface I inside the Vickers indentation when the load increased to 227.2 N. Until the final fracture occurred when subjected to a shearing load of 247.9 N, no obvious interfacial cracking was visible. As a matter of fact, the
microcrack nucleation locations were widely observed at dentin side, resin side and the interface, and the microcracks located at interface I could be considered as the sources to induce the continuous crack propagation and formation of main crack. Regarding Interface II, the resin inside the indentation imprint experienced extrusion cracking, extrusion release, planarization of extrusion deformation and generation of residual crack after fracture, successively. When subjected to a shearing load of 93.9 N, crack nucleation was also observed on the ZrO2 side. Initial interfacial cracking and indentation edge cracking were also observed when the load further increased to 160.8 N. On basis of 227.2 N, simultaneous interfacial cracking on both sides of indentation were observed, but the surficial rather than internal cracking was confirmed as the indentation bottom did not crack completely. Meanwhile, a circular arc crack profile inside the indentation imprint was observed, which demonstrated the brittle cracking of ZrO2 ceramic contributed to the bonding failure of resin-ZrO2 interface. Furthermore, when subjected to an equivalent shearing load of 247.9 N, the resin-ZrO2 interface completely cracked, accompanied with the falling of previously formed ZrO2 fragment. Therefore, the actual fracture process of ZrO2-resin-dentin specimen consisted of three processes, including initial interface cracking with mainly elastic extension, crack propagation with remarkable plastic flow and interfacial separation failure. Regarding the dentin-resin interface, although the prefabricated Vickers indentation defects with edge length of tens of microns has leaded to significant stress concentration effect, and plenty of multi-oriented microcracks nucleating inside the indentation imprint were visible, the interfacial cracking failure appeared at the resin-ZrO2 interface. Seen from the ultimate cracked interface, the competition between the Young’s modulus or hardness gradient of resin-ZrO2 interface and the stress concentration factor induced by Vickers indentation determined the crack propagation process and location of complete fracture failure.

Regarding the resin-ZrO2 adhesive interface, the interface separation process was quantitatively described by analyzing the crack width and depth evolutions as a function of shearing load. Seen from the shearing load responses of ZrO2-resin-dentin specimen as shown in figure 4(a), based on a shearing load of 93.9 N, multi-oriented microcracks were observed at various regions, the crack propagation behaviors under relatively larger shearing loads with range from 93.9 N to 200.5 N were taken into account. The crack width and depth evolution analyses were based on 10 double L-shape specimens with identical preparation process and loading conditions. As illustrated in figure 5(a), approximate linear increasing trend of crack width was obtained. The combined effects of prefabricated Vickers indentation and interfacial shearing load induced significant bonding cracking with crack width of 4.09 μm under a relatively lower shearing load of 93.9 N. As shown in figure 5(b), with increasing load ranging from 120.6 N to 200.5 N, the corresponding crack width presented a monotonic increasing trend with range from 6.04 μm to 11.69 μm. Through linear fitting, the linear correlation coefficient \( R^2 \) of width-load curve was calculated as 0.9953. With the aid of an optical microscope (Olympus DSX500) with three-dimensional imaging function and z-axis resolution of 0.01 μm, typical dimple-like crack region was selected to equivalently calculate the crack depth. Similarly, the approximate linear increasing of crack depth was described as shown in figure 5(c) with linear correlation coefficient \( R^2 \) of 0.9912. Meanwhile, although the observed microregions exhibited height difference with specific gradient, the crack depth was determined as the height difference between the average height of neighboring microregions along the interface and the lowest point of dimple-like crack. As shown in figure 5(d), with increasing load ranging from 93.9 N to 200.5 N, the corresponding crack depth also presented a monotonic trend with range from 3.72 μm to 10.33 μm.

### 3.2. Interfacial cracking width

Since the discontinuous fracture processes of compound interfaces were directly observed through \textit{in situ} shearing tests under microscope, and the resin-ZrO2 interface has already completely fractured prior to the
dentin-resin interface, the ultimate morphology of not yet completely fractured dentin-resin interface was characterized as illustrated in figure 6. As a matter of fact, although macroscopic cracks were not directly observed at the interface, the scanning electron microscopy images also indicated intermittent interfacial cracks [40]. The width of the widest microcrack was measured as 1.4 μm. Meanwhile, the location of the surface crack initiation did not originate from the dentin-resin interface but dentin’s interior. The minimum spacing between the dentin-resin interface and intermittent cracks, which were approximately parallel to the interface, was calculated as 2 μm. Furthermore, although the Vickers indentation with edge length of tens of microns has induced significant stress concentration effect, the crack propagation path intersected the indentation edge at a clear point and stopped continuous extension inside the indentation imprint, which demonstrated that the cracks nucleated from the dentin’s interior under quasi-static shearing stress. The complete fracture of resin-ZrO₂ interface prior to the dentin-resin interface was attributed to the acid etching of dentin [41–43] and significant gradients of interfacial mechanical properties, as a 2.2 times modulus ratio and 2.4 times hardness ratio of resin-ZrO₂ to dentin-resin interfaces have been experimentally confirmed.

Figure 5. (a) Crack width evolution as a function of shearing load and corresponding (b) separation process of the resin-ZrO₂ adhesive interface, (c) and (d) illustrate corresponding depth evolution as a function of shearing load and crack depths of dimple-like crack regions.

Figure 6. SEM images of dentin-resin interface to confirm the location and width of cracks.
3.3. Interfacial morphologies
The microstructures of complete fractured resin-ZrO\textsubscript{2} interface were also analyzed to indirectly verify the effect of acid etching on the interfacial bonding strengthening effect. As shown in figure 7, because the bonding process of resin-ZrO\textsubscript{2} interface did not involve an acid etching treatment, both the resin and ZrO\textsubscript{2} surfaces manifested as approximately flat and smooth features, which weakened the interfacial bonding strength. The resin layer manifested as a general smooth shear surface under quasi-static interfacial shearing stress intersected with stripe shaped residual resin due to the locally inhomogeneous plastic flow of resin. Meanwhile, discrete residual resins were observed distributing on the ZrO\textsubscript{2} surface unevenly. Residual resins were mostly round or elliptical in shape with maximum single residual area of approximate 700 \(\mu\text{m}^2\) (diameter of 30 \(\mu\text{m}\)). With the aid of machine vision technology, through the chromatic aberration analysis and image recognition [44], the actual residual area of resin on ZrO\textsubscript{2} ceramic surface was less than 5\%, which directly caused the low stress cracking of resin-ZrO\textsubscript{2} interface. According to the previous research hypothesis, the researches above indicated that the \textit{in situ} experimental study was useful to investigate the problematic dentin interface restorations, and the interfacial difference in mechanical properties was verified to induce the bonding failure and discontinuous fracture behavior of ZrO\textsubscript{2}-resin-dentin compound interfaces. However, this study was unable to achieve the \textit{in situ} testing under actual oral environment. Meanwhile, considering that the cyclic loading caused by repeated chewing force would lead to interface fatigue failure, the \textit{in situ} fatigue testing of ZrO\textsubscript{2}-resin-dentin specimen would be benefit to reveal the micro failure mechanism and comply with the requirements of clinical scenario.

4. Conclusions
Discontinuous fracture behaviors of ZrO\textsubscript{2}-resin-dentin bonding interfaces were experimentally revealed through equivalent \textit{in situ} interfacial shearing tests. Controllable shearing load and thermostatic medium...
environment filled with artificial saliva were realized to carry out the in situ tests under approximate oral environment. Interfacial difference in mechanical properties induced the low stress fracture (from the external to internal) of resin–ZrO₂ interface. With regard to the dentin-resin interface, the acid etching treatment and relatively closer modulus or hardness differences between dentin and resin effectively restrained the interface cracking. Compared with bonding strengthening effect by acid etching of dentin, the low stress fracture and adhesion weakening of resin–ZrO₂ interface were mainly attributed to the significant differences in modulus ratio (19.63) and hardness ratio (34.66) of ZrO₂ to resin, which promoted the crack nucleation and contributed to the interfacial separation and ultimate bonding failure.

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