Original article

Development of dental inspection method: nondestructive evaluation of a dentin–adhesive interface by acoustic emission

Ryoma Ezaki^{a,1}, Atsushi Mine^{a,1,*}, Kazuhisa Sato^b, Ken-ichi Fukui^c, Keigo Kumada^b, Masahiro Yumitate^a, Shintaro Ban^a, Azusa Yamanaka^a, Mariko Matsumoto^{d,e}, Bart Van Meerbeek^c, Toshiyuki Hashida^b, Hirofumi Yatani^a

^a Department of Fixed Prosthodontics, Osaka University Graduate School of Dentistry, Osaka, Japan

^b Fracture and Reliability Research Institute, Graduate School of Engineering, Tohoku University, Miyagi, Japan

^c The Institute of Scientific and Industrial Research, Osaka University, Osaka, Japan

^d Department of Restorative Dentistry, Hokkaido University Graduate School of Dental Medicine, Hokkaido, Japan

^e KU Leuven (University of Leuven), Department of Oral Health Sciences, BIOMAT & UZ Leuven (University Hospitals Leuven), Dentistry, Leuven, Belgium

¹ Equal first-author contribution.

Abstract

Purpose: The state of adhesion between root dentin and a resin composite core material was inspected using acoustic emission (AE).

Methods: A total of 14 human incisors and premolars were used to prepare “no-adhesive group” and “adhesive group” specimens. For “adhesive group” specimens, a bonding agent was applied to root canal dentin. The entire post space was subsequently filled with a resin composite for both specimen groups. The prepared specimens were fixed onto a jig on which an AE sensor was installed. A zirconia ball was used for the impact test, and a vibration wave generated by the collision was measured by the system using an AE sensor. The obtained data were subjected to time–frequency analysis using analysis software (LabVIEW), and the relationship between the amplitude indicating the loudness and the frequency indicating the sound component was analyzed.

Results: Zirconia-ball collision tests using AE revealed differences between the groups with respect to the waveform of vibration waves transmitted to the root dentin through the root dentin–resin interface. The time–frequency analysis of the obtained data confirmed that multiple peaks were observed for each specimen in the no-adhesive group, whereas a single characteristic vibration peak was observed for all specimens in the adhesive group.

Conclusions: The state of the adhesive interface was successfully evaluated by AE. This demonstration is expected to lead to the development of a device that can detect problems at the bonding interface between the prostheses and tooth substances.

Keywords: Adhesive dentistry, Microcomputed tomography, Acoustic Emission, Mild type one-step self-etching adhesive, Dentin bonding durability

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1. Introduction

In dentistry, the detachment of restorations and prostheses because of marginal leakage and secondary caries is challenging. The reported causes of these two major problems include residual infected dentin [1–4], poor penetration of the adhesive into the teeth [5], elution of residual unpolymerized monomer [6,7], and degradation of the adhesive and/or tooth [8,9]. Physical factors include oral temperature changes and biting force, which create excessive stress at the adhesive–restoration and prosthesis interface [10–12]. In current clinical practice, however, no satisfactory method is available to detect problems with the adhesive interface over time after prosthetic restorations have been completed.

Non-destructive evaluation methods such as X-ray, microcomputed tomography (μ CT), and optical coherence tomography (OCT) have been used to evaluate such interfaces in recent years [13–17].

μ CT, which can image small objects with high spatial resolution, has been widely used in biomedical research for examining the structure and mineral content of bones and teeth. It is now widely used for various non-destructive evaluations. However, μ CT cannot image the interior of radiopaque materials such as metals, and it lacks sufficient resolution to evaluate the adhesion between teeth and restorations. OCT operates on the principle of optical interferometry, where near-infrared reflected light that passes through living tissue [18] is used to analyze the internal structure approximately 3 mm from the surface in real time [19]. However, this technique encounters problems such as the irregular reflection of near-infrared rays in the case of rough surfaces and the inability to analyze non-translucent materials [19]. Therefore, the adhesive interface with restorations cannot be analyzed in the oral cavity using μ CT or OCT.

Acoustic emission (AE) is a phenomenon in which elastic energy is released through plastic deformation or fracture of solid material, and then it propagates through the solid as sound waves [20]. The use of AE for analysis was first reported by Kaiser et al. [21] in 1953 and has since been used to evaluate the states of materials and mechanisms of failure. For example, AE has been used to evaluate the degree of deterioration of concrete structures based on the crack initiation process [22]. Alternatively, the mechanical properties of a material can be estimated using AE to detect crack initiation owing to fatigue in the material [23–25]. In the field of dentistry, AE has been used to

* Corresponding author at: Department of Fixed Prosthodontics, Osaka University Graduate School of Dentistry, 1-8, Yamadaoka, Suita, 565-0871 Osaka, Japan
E-mail address: mine@dent.osaka-u.ac.jp (A. Mine).

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detect interfacial delamination owing to polymerization shrinkage stress of a composite resin [13,26–29] and microcrack propagation at an adhesive interface [13,30]. The mechanical properties of a composite resin [31], a destruction process [32,33], and the initial and final fracture load values of metal-free crowns [34] have also been measured by AE. In such analyses, AE measures the strain within the material and the fracture process, combined with various destructive tests and non-destructive evaluations. However, it has not been used to evaluate the adhesion interface of restorations and prostheses after setting or luting. In the present study, AE was used to confirm the state of adhesion between root dentin and a resin composite for core build-up. Our null hypothesis is that differences in the state of adhesion cannot be detected by AE.

2. Materials and methods

2.1. Microleakage under μ CT

2.1.1. Preparation of Specimens (Fig. 1)

A total of six caries-free human incisors and premolars were selected for this experiment. The experimental protocol was approved by the Ethics Committee of the Osaka University Faculty of Dentistry (H26-E6). The teeth were decoronated using a low-speed diamond wheel saw at the cement–enamel junction under water cooling. Root canals were prepared using a K-file (K-file, MANI, Tochigi, Japan). The canals were shaped with a size 80 K-file applied to the working length and were obturated by a lateral pressurized root canal sealer (Canals N, Showa Yakuin Kako Co. Ltd., Tokyo, Japan) and gutta-percha points (GUTTA PERCHA POINTS, GC, Tokyo, Japan). After the canals were filled, they were temporarily sealed with stopping (Shofu Temporary Stopping, Shofu, Kyoto, Japan) and stored in 37 °C water for 24 h. The canals were then enlarged using low-speed preparation drills (Tokuyama FR drill for post preparation, Tokuyama Dental, Tokyo, Japan) with working lengths of 10 mm from the cement–enamel junction. Following preparation, the canals were pretreated with a 3% ethylenediaminetetraacetic acid (EDTA) solution (Smear Clean, Nipponika Yakuin Co., Ltd., Yamaguchi, Japan) for 2 min and a sodium hypochlorite gel (AD gel, Kuraray Noritake Dental, Tokyo, Japan) for 1 min. The canals were irrigated with distilled water. Subsequently, after being blown with air for 10 s, the specimens were sufficiently dried using several paper points (Color Code Paper Point, GC, Tokyo, Japan) until the points were visually determined to not be wet with water. The thus-prepared specimens were defined as the “no-adhesive group.”

Furthermore, for “adhesive group” specimens, a light-cure one-step self-etching adhesive system bonding agent (Clearfil Universal Bond Quick, Kuraray Noritake Dental) was used according to the manufacturer’s instructions for bonding to root canal dentin for 10 s. After the specimens were air-dried for 10 s, excess bonding agent at the bottom of the canal was removed using a paper point and then light-cured for 10 s using an LED curing light (Mini LED3, the maximum light intensity of 2200 mW/cm², Satelec, Mérignac, France). The entire post space of each group (i.e., adhesive and no-adhesive groups) of the specimens was subsequently filled with dual-cure resin composite core material (Clearfil DC Core Automix ONE, Kuraray Noritake Dental). The specimens were light-cured for 20 s from five directions (occlusal, buccal–lingual, and medial–distal sides). In this study, we did not insert the prefabricated post in order to focus on the analysis of the adhesion between the resin composite core material and the dentin.

2.1.2. μ CT observation (Fig. 2)

Nail varnish (Top Speed First Dry Nail Enamel C07 Revlon New York USA) was applied to the outer surface of the root and 1 mm outside the root dentin–resin interface to coat the outer areas of the root dentin–resin interface. After drying the nail varnish, 50 gf of silver nitrate reagent (Kishida Chemical, Osaka, Japan) was dissolved in 50 mL of distilled water to prepare a 50% silver nitrate aqueous solution, and each specimen was immersed in the aqueous solution for 24 h. The microleakage was measured by μ CT (R_mCT2, Rigaku, Tokyo, Japan) under the imaging conditions of a tube voltage of 90 kV, tube current of 160 μ A, resolution of 20 μ m, and a field of view of 10 mm ($n = 3$). The leakage site of silver

Fig. 1. Specimen preparation.

a: The crown was removed. b: The root canal was prepared and obturated. c: The root canal was enlarged. d: The post space was irrigated. e: The post space was dried with an air syringe and paper points. f: The adhesive was applied and light-cured. g: The post space was filled with dual-cure resin composite core material and light-cured.

Fig. 2. Zirconia ball impact test (left: illustration, right: actual photo).

a: specimen, b: AE sensor, c: zirconia ball.

*On J-STAGE, videos are available as Supplementary material.

nitrate was visualized three-dimensionally using image analysis software (TRI 3D BON, RATOC System Engineering, Osaka, Japan).

2.2. Nondestructive evaluation by AE

A total of 14 caries-free human incisors and premolars were used to prepare the “no-adhesive group” and “adhesive group” specimens ($n = 7$, Fig. 1). The protruding resin composite core material was polished with water-resistant abrasive paper (600#, Sankyo Rikagaku, Saitama, Japan) such that the resin composite core material surface coincided with the cut surface at the cement–enamel junction of the specimens. The prepared specimens were fixed onto a jig on which an AE sensor (AE-900S-WB, NF circuit design block, Kanagawa, Japan) was installed (Fig. 2). A 0.4-gf, 5-mm-diameter zirconia ball (YTZ® ball, Nikkato, Osaka, Japan) was used for the impact test. The acrylic tube used to slide the zirconia ball was adjusted to an angle of 45° relative to the resin composite core material. The starting point of the fall of the zirconia ball was set such that the vertical fall distance was 50 mm (Fig. 2, Supplementary Video 1). The condition settings related to the zirconia balls were based on the following. First, we used zirconia balls (YTZ® balls, Nikkato, Osaka, Japan) as a standardized material, as they are generally used as dental material and have good impact resistance. We also selected a size that fits within the diameter of the resin post. Second, the conditions of weight, drop distance, and gliding angle of the zirconia balls were determined in preliminary experiments. If the impact of the falling zirconia balls is too large or too small, more vibration waves are transmitted to the outer surface of the specimen than the vibration waves transmitted from the resin post through the root dentin to the interior of the specimen. Thus, it may not be possible to accurately detect the vibration wave transmitted inside the specimen. A vibration wave (i.e., impact sound) generated by the collision was measured by the system using an AE sensor, preamplifier (9917, NF circuit design block), discriminator (9922, NF circuit design block), and oscilloscope (WaveSurfer 424, Teledyne LeCroy, Tokyo, Japan). The principle of AE is illustrated in Fig. 3. The obtained data were subjected to time–frequency analysis using analysis software (LabVIEW, National Instruments,

Texas, USA), and the relationship between the amplitude indicating the loudness and the frequency indicating the sound component was analyzed.

3. Results

3.1. Microleakage under μ CT

The μ CT images of the gaps and air bubbles in the root canal dentin–resin interface exhibited no difference between the adhesive group and the no-adhesive group before the microleakage test (Fig. 4). No difference was observed even in the specimens subjected to the microleakage test using silver nitrate (Fig. 5). However, in the three-dimensional image of a setting with a silver nitrate threshold, silver nitrate (i.e., a gap) was observed at the dentin–resin interface from the whole crown side to the apex side of the no-adhesive group (Fig. 6).

3.2. Nondestructive evaluation by AE

The waveform of vibration waves transmitted to the root dentin through the root dentin–resin interface as a result of a zirconia-ball collision test using AE differed between the adhesive group and the no-adhesive group (Fig. 7). The waveform of the no-adhesive group attenuated gradually as the amplitude was repeatedly increased and decreased every 3 ms. In contrast, the waveform of the adhesive group attenuated smoothly from the maximum amplitude. The time–frequency analysis of the obtained data revealed that the highest peak frequency was different for each specimen in the no-adhesive group, and multiple peaks were confirmed for each specimen (Fig. 8). However, a single characteristic vibration peak was observed for all specimens in the adhesive group (Fig. 9).

4. Discussion

In recent years, μ CT has been widely used for non-destructive evaluation in various fields [14]. μ CT is a computer-assisted three-dimensional reconstruction system that can create slice images in any direction to provide information about the internal structure. In the research on dental materials, μ CT is used for various purposes, such as the analysis of polymerization shrinkage of dental adhesives [15], quantification of the internal porosity of composite resin restorations [16], and confirmation of the presence of an adhesive interface and internal gap [17]. However, its resolution is limited, precluding detection at the submicron level [13]. In the present study, no clear difference was observed in the appearance of bubbles and gaps at the dentin–resin adhesive interface via simple μ CT imaging. Therefore, we compared the μ CT images before and after the microleakage test using silver nitrate. More silver nitrate was found to have penetrated the dentin–resin bonding interface on the coronal side of the no-adhesive group than the interface on the coronal side of the adhesive group only when the threshold of silver nitrate was set and three-dimensional imaging was performed.

Notably, a difference between the no-adhesive group and the adhesive group that was detected by simple imaging with μ CT was detected as a difference in vibration waves under AE. Therefore, our null hypothesis that the difference in the adhesion state cannot be detected by AE was rejected. Although μ CT is useful for non-destructive observation, its resolution is limited and clinical application is difficult at present [27]. In addition, because X-rays cannot penetrate metal, an adhesion interface in a metal crown cannot be evaluated. Moreover, X-ray exposure is unavoidable when evaluating the adhesive interface using μ CT. In contrast, AE can be evaluated irrespective of the material because it detects and evaluates the vibration wave of the object; it can, therefore, be applied without exposing the patient to X-rays.

AE is a non-destructive evaluation method that evaluates vibration waves whose peculiar characteristics depend on the density and mass of the material. The root dentin and resin composite core material evaluated in the present study also have unique vibration waves.

Fig. 3. Principle of conventional AE.

An oscillatory wave is generated via an impact induced by applying an external force to the specimen. The propagated vibration wave changes in the place where the defect inside the specimen changes, and the vibration wave is detected by the AE sensor. The existence of internal defects is confirmed by the difference in the vibration waves.

Fig. 4. Micro-CT images before the AgNO_3 infiltration. D: dentin; RC: resin for abutment construction.

Fig. 5. Micro-CT images after the AgNO_3 infiltration. D: dentin; RC: resin for abutment construction. Arrow: penetration of AgNO_3 .

The vibration wave differs substantially between the case when two substances vibrate as one object by adequate bonding and the case when the bonding interface is insufficient and the two substances vibrate independently. In the present study, this clear difference was evaluated in the no-adhesive and adhesive groups by AE. The first peak of the obtained vibration wave was used to evaluate the difference in vibration. The first peak of the elastic wave is not affected by the resonance sound, and this effect is a factor influencing the vibration characteristics of the

Fig. 6. Three-dimensional image of the AgNO₃.

Fig. 7. Vibration waveform obtained by zirconia-ball impact test using AE. a: no-adhesive group; b: adhesive group. *:Starting point of vibration wave (collision sound).

materials. As a result of the zirconia-ball impact tests, two waveforms (i.e., the no-adhesive group and the adhesive group) of the vibration wave transmitted from the resin composite core material to the root dentin through the root dentin–resin adhesive interface were distinct. Multiple characteristic vibration peaks were observed in the time–frequency analysis of all specimens in the no-adhesive group. In contrast, in the analysis of all specimens in the adhesive group, a single characteristic vibration peak was observed, except for the weaker peak in the frequency band near 75 kHz. The observation of multiple peaks indicates that multiple substances vibrated because the root dentin and the resin composite core material were not bonded in the no-adhesive group. The occurrences of these multiple peaks might have been affected by the morphology of the specimen. In the no-adhesive group, multiple peaks were randomly generated by vibrating multiple substances with various morphological characteristics. In contrast, in the adhesive group, a resonance sound was generated in the individual substance and thus vibrated. This was considered to be a weak peak in the frequency band near 75 kHz and 750 kHz. In the adhesive group, the root dentin and the resin composite core material exist as a single substance because of adhesion; peaks with vibration characteristics thus became a large single peak.

The present study revealed that the state of the adhesive interface can be evaluated by AE using zirconia-ball impact tests. We successfully distinguished between the no-adhesive group and the adhesive group by observing the differences in the vibration of the materials. However, AE is difficult to apply in clinical applications because it requires the application of an external physical force with good reproducibility. Furthermore, the stimulus caused by this force may impose a load on the test object, leading to the destruction of the adhesive interface. In the future, solutions to these problems and the miniaturization of devices are expected to lead to the development of a device that can detect problems at the bonding interface between prostheses and tooth substances. If the difference in the adhesive state can be evaluated by detecting the difference in vibration waves by AE devices, it may be possible to determine the need for retreatment clinically and non-destructively.

Fig. 8. Results of time–frequency analysis of zirconia-ball impact tests using AE in the no-adhesive group.

Fig. 9. Results of time–frequency analysis of zirconia-ball impact tests using AE in the adhesive group.

5. Conclusion

1. The 3D image of micro-leakage observation by μ CT revealed a gap formation at the bonding interface without using adhesive.
2. AE can detect a difference in the state of adhesion between root dentin and resin composite core material.

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Conflict of interest statement

None

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