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Review Article

The microtensile bond strength test: Its historical background and application to bond testing

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SUMMARY

Microtensile bond strength (μTBS) test was introduced in 1994. Since then, it has been utilized profoundly across many bond strength testing laboratories, making it currently one of the most standard and versatile bond strength test. Although it is a static and strength-based method, together with the morphological and spectroscopic investigations, it has been contributing immensely in the advancement of dentin adhesive systems. μTBS test has a greater discriminative capability than the traditional macro-shear bond test. During the early stage of its development, the authors predicted that this testing method would enable evaluation of the adhesive performances of resins to excavated carious or sclerotic dentin and the regional bond strengths of various portions of the cavity. In addition, they also stated the possibility of comparing the long-term stability of resin adhesion at various portions of the cavity walls on teeth extracted at various times after insertion of bonded restorations. In this review, we discussed the historical background, inception and the application of the μTBS test and proposed directions for further improvement of this testing method.

1. Introduction

New concepts in operative dentistry proposed by Fusayama [1] opened up new treatment strategies by means of advanced knowledge in cariology and introduction of dental adhesives. Restorative principles proposed by GV Black [2] such as “Extension for Prevention”, “Retention Form” and “Resistance Form” have been modified and/or replaced by “Minimal Invasive Dentistry” [3]. Minimal invasive (MI) approach of cavity preparation basically aimed to minimize the reduction of tooth substances to preserve maximum sound tooth structure.

Enamel adhesion introduced by Buonocore [4] has been regarded as reliable and characterized by strong bonding between enamel and adhesive resin (Fig. 1). The mechanism of enamel adhesion entails penetration of adhesive monomers into acid etched enamel prisms, enveloping superficial apatite crystals [5]. Extensive studies by Yoshida et al. [6,7] showed direct evidence of chemical bonding of molecules with functional groups using synthetic hydroxyapatite. Recent studies suggested that the hydrolytically stable calcium salt formation of “nanolayering” at the adhesive interface could contribute to the durability of the bonds [8,9] and enhance the bond strength [10] when using the adhesives containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) as a functional monomer. The concept of “nanolayering” appears to be included in that of the “nanointeraction zone” which was predicted and proposed by Koshiro et al. in 2006 [11]. They claimed that the nature of the adhesion through hydroxyapatite was based on the nano-level chemical interactions between the functional monomers and hydroxyapatite *per se*. However, it is still not clear whether “nanolayering” can fully envelope the hydroxyapatite *in situ* which is highly susceptible to acid attack within the oral environment. Further studies are needed to clarify the clinical significance of “nanolayering” between the resin and the hydroxyapatite of the tooth.

The first remarkable impact of adhesive technology on clinical dentistry was the introduction of the direct bonding system (DBS)

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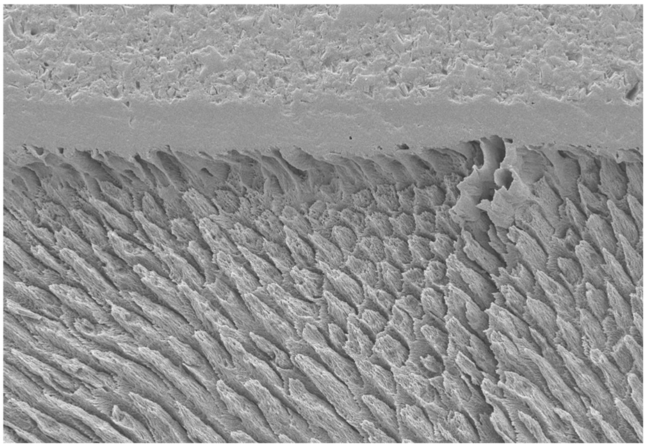

Fig. 1. Resin-enamel interface (×1000) under FESEM.

proposed by Miura et al. [12]. Before DBS, orthodontists in the clinics had to perform the time-consuming and technique sensitive procedures and patients had to endure uncomfortable treatment time [13]. In those days, the orthodontists used to place metal bands with welded brackets on the teeth circumferentially, for which, they needed to create the interproximal space in advance to ensure the width of the band material. Moreover, the universal use of orthodontic bands on all the teeth had potential risks of gingival trauma and caries under or near the bands [13]. With the introduction of DBS, brackets could be placed directly on the tooth enamel, which enabled the orthodontists to gradually replace the bands with brackets, making the bands almost nonexistent in the current clinical practice.

Bonding to dentin was challenging, because dentin is a composite material approximately with 50 vol% of mineral phase, 30 vol% of collagen and 20 vol% of water [14]. Bonding strategy to dentin focuses on both mineral phase and organic phase (mainly collagen) under the moist environment. Moreover, most of the clinical substrates are covered with smear layers (Fig. 2), which act as barriers against the penetration of adhesives' molecules into tooth substances [15–20]. In clinical situations, removal or modification of the smear layer is therefore essential to create the hybrid layer to ensure a strong bond between the resin and dentin [21–24].

2. Before the inception of microtensile bond strength (μTBS) test

The first marketed "total-etching (etch-and-rinse)" adhesive was Clearfil Bond system-F by Kuraray Co. (Tokyo, Japan) in 1978. Fusayama et al. [25] tested its bond strength to 40% phosphoric acid etched and unetched dentin. They found that the bond strength was significantly increased when using etching mode (i.e., approximately 2 MPa for unetching vs. approximately 6 MPa for etching mode). From the current point of view, their dentin bond strength values were very low. Moreover, testing the unetching mode for dentin bonding seems to be unusual since the product was not a self-etching dentin bonding agent. Nonetheless, it was the first marketed product claiming to achieve dentin adhesion. Moreover, at that time Fusayama introduced "total etching" technique, which was resisted by the US and European clinicians, since they were concerned about the probable pulpal irritation that could result from the bacterial invasion or penetration of chemical ingredients through the dentinal tubules opened up by acid etching [26,27]. In 1982, Inokoshi et al. [28] performed the histopathological investigation of pulpal response to Clearfil Bond system-F using dogs. When using total-etching technique followed by the application of the adhesive, pulpal responses were slight and there was no bacterial penetration through the cavity wall and dentinal tubules.

Although the total-etching proposed by Fusayama was "dry bonding" in nature, which required air blowing after acid etching and would collapse the exposed demineralized dentin [29], the technique spread quickly in Japanese clinical practice without causing noticeable clinical complications. The concept of adhesive cavity preparation proposed by Fusayama [1] endorsed preservation of the inner carious dentin which is less permeable because of the presence of sclerotic/transparent dentin. This might be one of the reasons for less clinical complications caused by total-etching technique despite of the rapid spread of adhesive restorations following the introduction of the first adhesive system.

Since the advent of the adhesive resin composite restoration in late 1970's, there is a persistent demand for the improvement of adhesive systems [1]. Research and development (R&D) of the new adhesive systems had been boosted in Japan. Research focuses of the academic sectors were also shifted from cast restorations to adhesive restorations [30]. During that period, many researchers and R&D persons had been using extracted human teeth to evaluate the bond strength of the adhesives [25,31,32]. However, extracted human teeth became increasingly difficult to obtain due to the progresses in preventive and tooth saving conservative dental treatments. Nakamichi et al. [33] suggested the use of bovine teeth as a possible substitute for human teeth in testing the bond performances of adhesives. Although the mechanical and morphological properties of bovine teeth are slightly different from extracted human teeth [34], they are still useful for R&D of new adhesive systems or screening bonding tests in pilot studies [35,36].

Bond strength tests have been evaluated in tension or shear modes. Tensile bond strength test had been employed in many reports since the early stages of the adhesion studies [25,31,37,38], though it required special testing jigs and meticulous testing procedures or a unique set-up. Although shear bond strength test generated non-uniform stress concentration at the edge of the bonded interface during testing [39,40], it also gained its foot hole in bond test studies [41–45], because this method was easier to

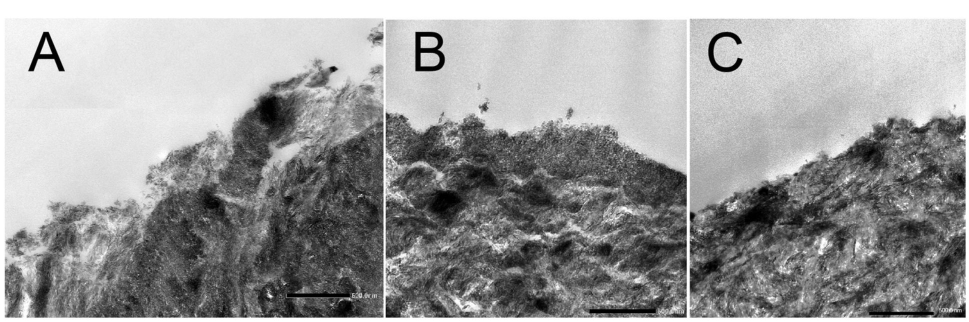

Fig. 2. Smeared and fractured surface of dentin under TEM, showing: each bur as 500 nm; (A) smear layer created with 600-grit SiC paper; (B) smear layer created with regular diamond bur; and (C) fractured smear-free surface.

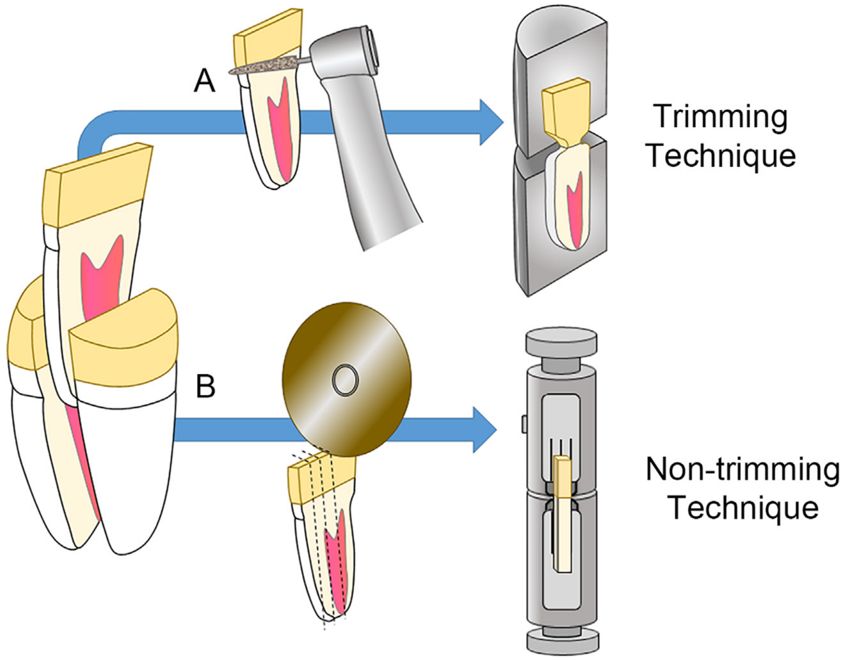

Fig. 3. Schematic representation of specimen preparation for microtensile bond strength test, showing: (A) trimming technique; and (B) non- trimming technique.

apply for testing the bonded interfaces compared to tensile bond strength test.

During 1980's, bond tests using shear or tensile modes were enough to evaluate the new adhesive systems available in the market [46]. Since the introduction of the primer for adhesion [47,48], bond strength values tested by means of conventional testing methods increased and reached the plateau, the measured values being approximately 18–20 MPa [46]. In other words, it was difficult to find significantly different bond strength values when evaluated using conventional shear or tensile bond test. Moreover, dentin cohesive failures with relatively lower bond strength (i.e., less than 10 MPa) were frequently observed [49–53], though the reason for cohesive failures in dentin in such low bond strengths was not clearly understood at that time.

3. Inception of the μTBS test

The first specimen-shape used for the μTBS test [54] was the hourglass shape, which duplicated the specimen shape fabricated by Akimoto [55] and was employed for the study to test the tensile strength of mineralized and demineralized dentin [56]. Akimoto's study was also focusing on measuring the tensile strength of a plane material, i.e. mineralized or demineralized dentin. Therefore, μTBS test was introduced with similar shaped specimens, though the resin/dentin interface was located at the narrowest portion of the specimen, aiming to increase the stress concentration at the adhesive interface to induce the catastrophic rupture of bonding (Fig. 3).

Interestingly, the tensile bond strength was dependent on the surface area for bonding [54]. Results of the tensile bond strength were plotted as a function of bonded surface area and they fitted into a decreased inverse logarithm curve with greater incidence of cohesive failures in dentin when bond strength was measured with greater surface area [57]. According to the Griffith's defect theory [58], the tensile strength of a material decreases with increasing specimen size when testing uniform materials in tension. This result may be due to the distribution of defects in the material because a larger specimen probably contains more defects compared to smaller specimens. Though Griffith's theory was developed for uniform brittle materials, it may be applicable for tensile bond testing as well because of the observed surface area dependency noted in measuring μTBS [54 Sano, 1994]. The aforementioned study demonstrated the usefulness of a new tensile bond test using small surface areas, which showed more frequent adhesive failure at the bonded interface with higher bond strength compared to the conventional bond tests utilizing larger surface area for boding, which sometimes showed cohesive failure within dentin at less than 5 MPa [50].

In the μTBS test introductory paper [54], the authors predicted that it would be possible to evaluate the adhesive properties of resins to excavated carious or sclerotic dentin and the regional bond strengths of various portions of the cavity employing the μTBS test technique. They also stated the possibility of comparing the long-term stability of resin adhesion at various portions of the cavity wall using the μTBS test on teeth extracted at various times after insertion of bonded restorations. Moreover, they requested for the improvement of the testing method using surface areas even smaller than 1 mm^2 to permit regional evaluation of bond strength within complex cavity preparations rather than on flat surfaces.

Originally, μTBS test was performed using a gripping Bencor Multi-T (Danville Engineering Co., USA) apparatus attached to a universal testing machine [54]. From the original testing design, other apparatus variations were created and tested, such as, portable testing machines and various gripping devices [59]; and test variations and parameters have been studied [59–62]. Compact and portable testing machines are relatively low costing, which made μTBS test an accessible research resource and a popular method in adhesive dentistry.

Following the introduction of μTBS test [54], review articles [57,59,63] concerning the technique were published, which helped the researchers to employ the test for better understanding of tooth adhesion. As far as the data processing of the μTBS test results is concerned, several reports are available [59,64–69]. When conducting the μTBS test, these reports are aiding the researchers in their research planning and preparation and subsequently in publications.

4. Application of the μTBS test

More than thousand publications are available when a web searching for the μTBS test [70,71] is performed. The test itself together with the morphological and spectroscopic investigations has been contributing to improve resin/dentin adhesion and has proven to have a greater discriminative power than the traditional macro-shear test [30,46,57,63,68,72,73]. The μTBS test is currently considered as a versatile and standard bond strength testing method, although the interfacial fracture toughness testing is more valid than this testing [59,67].

As already mentioned, originally, the specimen of the μTBS test (Fig. 3-A) was trimmed to hourglass shape with rectangular cross-sectional shape (trimming technique) [54]. During the development stage of the test, careful trimming was performed using superfine diamond-points not to provide extra stress to the bonded interface and to reduce the creation of visible pre-notch which might create the origin of the crack propagation. Modification of the μTBS test was done to clarify the occurrence of large variations in bond strength values when serial sections were performed (Fig. 3-B) [74]. Basically, the modified testing method employed stick shaped specimens with 1 mm × 1 mm cross-sectional area (Fig. 3-B) (non-trimming technique). In this paper, the authors also stated that the largest differences between bond strength values were shown to be related to the technique rather than to the material, and the results indicated that resin-dentin bonds may not be as homogeneous as was previously thought. At that time, the impact of the dentin's regional variability and depth on the bond strength was not clear. The effect of the dentin depth on the μTBS will be discussed later in this section.

Theoretically, the stress distribution of cylindrical cross-sectional specimens at the resin-dentin interface should be more uniform than that of rectangular cross-sectional specimens when performing the μTBS test. Many reports are available using cylindrical cross-sectional specimens for the μTBS test [75–79]. Although the theoretical background of the specimens with cylindrical cross-section appeared to be desirable for the μTBS test, the fabrication of the specimen required special equipment and needed more time compared to that of non-trimming technique.

Phrukkanon et al. [80] reported interesting results. They compared the influence of cross-sectional shape and surface area on the μTBS using cylindrical cross-sectional specimens and specimens fabricated by means of trimming technique. According to the results of the study, cross-sectional shape had little effect on bond strength. Interestingly, bond strengths obtained with non-trimming technique were similar to those of specimens prepared with trimming technique [74]. Based on these findings, it can be concluded that μTBS values that are obtained by any of the three methods, i.e., trimmed specimens, non-trimmed specimens and specimens with cylindrical cross-section, can be similar. Among them, specimens being made in non-trimming technique could be the easiest and the least technique sensitive. This should be the main reason why most of the current researches of the μTBS test are employing non-trimming technique.

Many substrate related factors can affect the short-term μTBS values of adhesives to sound or altered flat dentin [81], such as, aged dentin [82–84], caries-affected dentin [85–90], sclerotic dentin [91–94], dentin's hydration status [95] and depth. The following sections will be focusing on further discussions on bonding to various depth or regions of sound dentin.

Depth of dentin is indirectly expressed by remaining dentin thickness (RDT). RDT as a parameter that affect *in vitro* bond strength was widely evaluated even before the introduction of the μTBS test [96–99]. At that time, it was generally accepted that deep dentin showed lower bond strength compared to superficial dentin [54]. Suzuki and Finger [96] reported that the area of sound dentin available for bonding was one of the main factors having influence on the tensile bond strength. Prati and Pashley [100] demonstrated that moist nature of deep dentin might dilute or precipitate the ingredients of adhesive and interfere with its proper curing and adhesion to the dentin.

After the introduction of the μTBS test, many dentin bonding studies were published with regard to the dentin depth or regional variations [101–106]. Pereira et al. [101] evaluated the influence of intrinsic wetness on regional bond strengths of two step self-etching and two step etch-and-rinse adhesive resins to different regions of dentin using three groups, evaluating the effects of no pulpal pressure, pulpal pressure of 15 cm H_2O and dentin dried overnight in a desiccator. They found that the self-etching adhesive system did not exhibit significantly different regional bond strengths, whereas the etch-and-rinse adhesive showed significant decrease in bond strength at the pulp horn region. Although the smear layer on dentin was created with 600-grit silicon carbide paper, the smear layer and the smear plug could block the additional dentin wetness when using the self-etching adhesive system. On the other hand, the etch-and-rinse adhesive employed in the study completely dissolved the smear layer and smear plug leading to the poor adhesion at the pulp horn region. According to the results of this study, the self-etching system appeared to block the intrinsic wetness because of its weak acidity and the etch-and-rinse adhesive system could create potentially weak regions of bonding on the dentin surface.

Recent studies demonstrated controversial results regarding the effect of dentin depth on bonding. According to the reports of Ting et al., [105,106] the bond strengths of the tested one-step self-etch adhesives were affected by RDT but the bond strength of Clearfil SE Bond, a two-step self-etch adhesive was independent of RDT. Yoshikawa et al. [104] reported that Clearfil SE Bond showed an inverse correlation between μTBS and RDT, whereas, Single Bond showed no significant difference in μTBS for any RDT. Pegado et al. [103] demonstrated that bond strength obtained with superficial dentin was significantly higher than that of deep dentin for all the adhesive systems tested (i.e., Adper Single Bond 2; etch-and-rinse, Clearfil SE Bond; two step self-etching, and Futurabond; one step self-etching). These controversial results were in agreement with the study that stated that the resin-dentin bonds might not be as homogeneous as was previously thought [74]. Thus, the depth or region of dentin provided us a large scatter in the bond strengths and has been proven to have significant impacts on bonding outcomes.

As mentioned before, dentin depth or dentin regions are significant factors in determining the μTBS strength of sound dentin. Therefore, additional care should be taken when conducting the μTBS test using flat normal dentin surfaces. Permeability through such flat dentin has regional variations, which affects the regional bond strengths from a single tooth [101,102,107–110] and can mimic the *in vivo* conditions for bonding. Many embedded specimens reported to have relatively dry pulp chambers [57,103]. In order to avoid drying of the pulp chamber of extracted tooth, non-embedded specimens with positive and/or zero pulpal pressure is desirable [63,101,111,112].

Technically, to perform the μTBS test using deep dentin as a substrate is difficult, especially in the case of RDT being less than 1 mm. Therefore, bonding and filling the pulp chamber with resin composite before cutting into sticks is a useful measure to ensure proper stick length for fixation to the jig [103]. However, this procedure creates relatively dry dentin specimens and excludes the effects of intrinsic water from the pulp chamber. Fig. 4 has been designed to show the proper method for measuring the μTBS of deep dentin. As is shown in the figure, resin composite build up for the μTBS testing must be done before filling the pulp chamber with resin composite. Another important procedure for specimen preparation is to

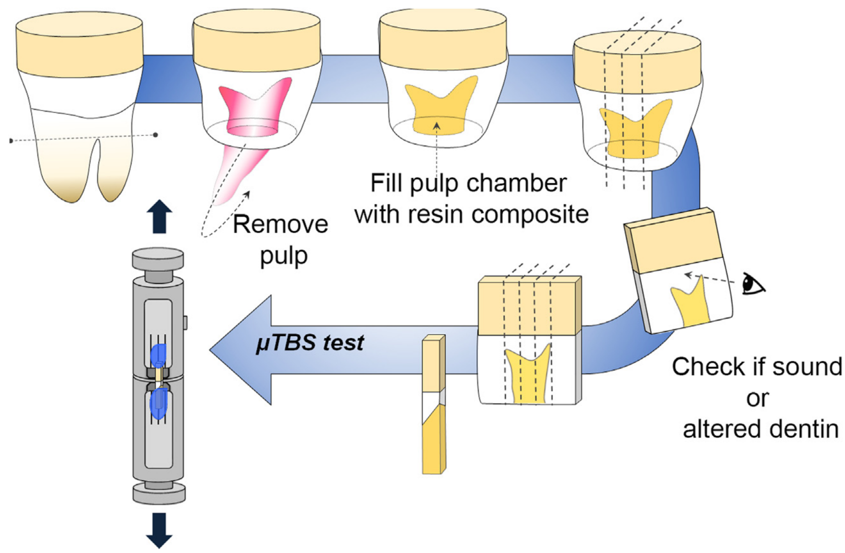

Fig. 4. Schematic representation of the procedure for measuring the microtensile bond strength (µTBS) of deep dentin.

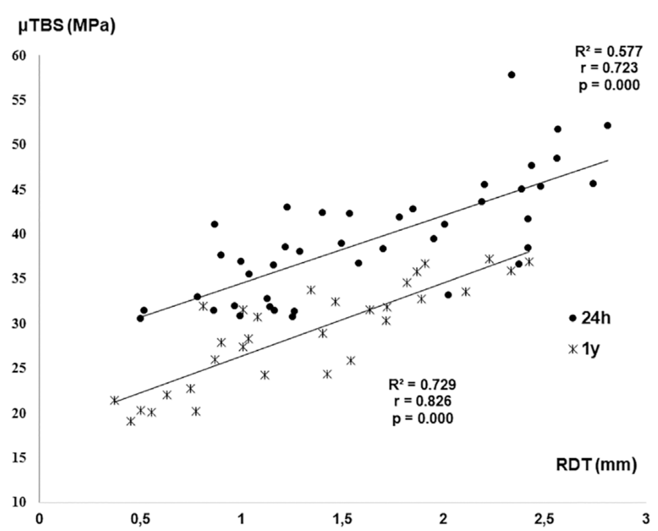

Fig. 5. Relationship between microtensile bond strength (µTBS) and remaining dentin thickness (RDT) as shown after 24 h and 1 y water-storage by a one-step self-etching system.

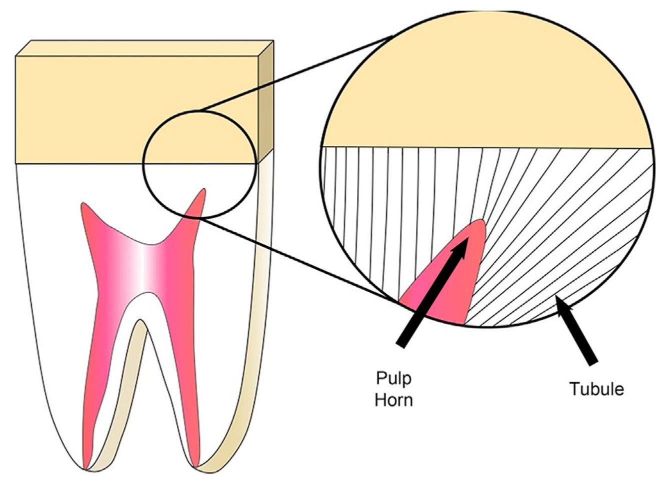

Fig. 6. Schema showing the proximity between the pulp horn and the prepared dentin surface. Dentinal tubules are radiating from the pulp to the prepared dentin surface.

perform careful examination of the slabs to determine whether the region to be tested is of sound dentin or altered dentin. Using this specimen preparation procedure, correlations between µTBS and RDT after 24 h and 1 y water-storage were analyzed using a one-step self-etching system and bond strength of very deep dentin (i.e., RDT < 0.5 mm) was also successfully evaluated (unpublished data; Fig. 5).

Deep dentin [103–106] and pulp horn region [101,102] are similar in that both show low bond strengths. Grinding the crown of molar teeth exposes flat dentin surfaces that can be categorized into central, peripheral and pulp horn region [101], the pulp horn regions are generally regarded as deep dentin. When making approximately 1 mm thick slabs perpendicular to the exposed dentin surface, lateral view of the slab shows that the pulp horns are very close to the exposed surface, meaning actually deep dentin. Dentinal tubules spread radially from the pulp horn to the prepared dentin surface with slight curvature (Fig. 6). The area of observable pulp horn regions on the exposed flat dentin surface have measurable size, which can partially or fully involve 1 mm × 1 mm cross sectional area for adhesion resulting in a large scatter of the obtained bond strength. This might also be the reason of the lacking of the homogeneous bond strength values [74]. Therefore, when calculating the mean value of a single tooth [59], avoiding the adverse effect of pulp horn regions should be of crucial importance.

5. Considerations of clinical substrates and future directions

The big advantage of the µTBS test is that the researchers can focus on clinically relevant substrate with three-dimensional surfaces. Sound and caries-affected dentin are important substrates for bonding in the clinical situation. Fusayama and his coworkers [1] proposed the two layers of carious dentin: outer carious dentin (caries-infected dentin) and inner carious dentin (caries-affected dentin) [113–115]. Caries-affected dentin is partially demineralized [116,117] and is believed to have a potential to remineralize [1,118]. Preservation of caries-affected dentin is therefore important in the clinical situation. However, caries-affected dentin is different in morphological, chemical and physical characteristics from sound dentin. Dentin hardness is one of the criteria in diagnosing dentinal caries [116,117,119].

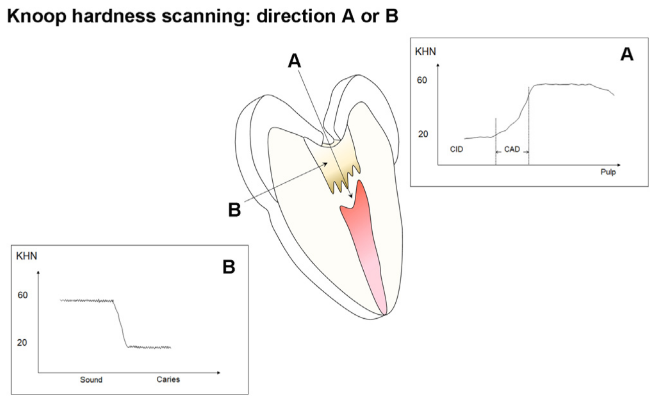

Fig. 7. Knoop hardness scanning of mid coronal dentin caries, performed (A) along the long axis of the carious lesion; and (B) perpendicular to the lateral surface of the carious lesion (tubule direction). CID: Caries-infected dentin; CAD: Caries-affected dentin; KHN: Knoop hardness number.

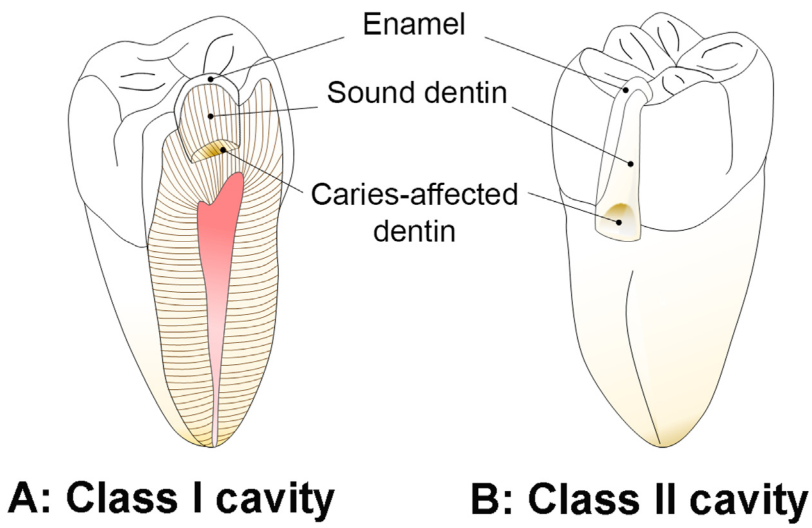

Fig. 8. Schema showing: (A) Class I cavity preparation; and (B) Class II cavity preparation for adhesive restorations based on Minimal invasive (MI) approach.

Sano [119] performed multiple scanning for Knoop hardness determination of mid coronal lesions of dentinal caries (Fig. 7). When the scanning was performed along the long axis of the carious lesion from the center of the caries-infected dentin to the pulp (Fig. 7-A), Knoop hardness number (KHN) of caries-infected dentin (CID) was less than 20–25, then the number increased gradually until it reached approximately 60 at the caries-affected dentin (CAD), followed by the plateau part of approximately 60, and then decreased gradually to the pulp. In this direction, thickness of the caries-affected dentin was measured approximately between 1000 μm to 1500 μm [119]. On the other hand, when the Knoop hardness measurement was performed perpendicular to the lateral surface of the carious lesion (tubule direction), it demonstrated different results (Fig. 7-B). Sound dentin recorded approximately 60 KHN and then KHN abruptly dropped to approximately 20 KHN. In this graph, the transition zone which corresponds to the CAD is very thin (less than 50 μm). Clinically, it is very difficult to preserve this thin layer often resulting in exposure of the pulp, whereas it is easy to preserve caries-affected dentin when excavating along the long axis of the carious lesion because of its thickness [119].

Fig. 8-A illustrates a Class I cavity preparation for a mid-sized coronal caries based on MI concept. Outline of the cavity is placed on the enamel with which adhesion is reliable. Cavity wall exposed by excavation is mostly in sound dentin along with the dentinal tubules, whereas the cavity floor being observed is caries affected dentin. When considering Class I resin composite restorations and different bonding situations, such as bonding to sound enamel, bonding to sound dentin perpendicular to the tubule direction, and bonding to caries affected dentin under the presence of shrinkage stress of resin composite, so far, no reports are available focusing on the 3D bonding to different substrates. With regards to restoration of Class II cavity, the proximal margin should be in sound dentin, sclerotic dentin, or altered or caries-affected dentin (Fig. 8-B). Detailed bonding characteristics to such dentin are not yet clear. Further studies are needed to disclose the bonding characteristics to the cavity wall with regard to above mentioned Class I and Class II cavities.

The microtensile bond test is a very useful tool for bonding research. However, pretesting failure is still occurring as a significant problem for the testing when using adhesives with low bond strength. Further improvements are necessary to overcome this issue.

Scientific field of dental science

Operative Dentistry.

Conflicts of interest

None.

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