Fracture Aspects of Resin-Dentin Bonding in Non-trimming Microtensile Test

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Comparative studies on resin-dentin bond strength and failure mode were performed between the conventional tensile test and the microtensile test with non-trimming small specimens, 1×1 mm in cross-section, for two brands of dentin bonding systems. The fracture surface of the conventional large specimen showed a catastrophic cohesive failure in dentin at its center and a lesser adhesive failure, suggesting that the whole failure was due to the development of some major cracks. The non-trimming microtensile test showed significantly larger average bond strength with markedly larger standard deviation and significantly larger fraction of adhesive failure than the conventional test. Some small specimens were extremely strong and some were weak according to the heterogeneous distribution of tight bonding and defective or deficient bonding over the whole dentin surface. These results suggest that the non-trimming microtensile test may potentially provide more realistic aspects of resin-dentin bonding than the conventional bulk specimen.

Key words: Resin-dentin bonding, Non-trimming microtensile test, Failure mode

INTRODUCTION

Since the introduction of dentin primer and the improvement of the dentin bonding system for composite resin restoration, the adhesion strength has greatly increased and the failure mode has tended to change from adhesive failure to cohesive failure within dentin by the use of the conventional tensile testing method¹ ². Since only one specimen can be prepared from one tooth, either a human tooth or a bovine tooth, in the conventional tensile test, it is necessary to prepare many teeth to gain more information or measurements for a reliable analysis. As in the study by Retief³ that reported human teeth can provide more realistic information than bovine teeth, it is better to use human teeth for testing. Unfortunately, however, it is not easy to obtain many human sound teeth.

A microtensile testing method was therefore developed, in which a resin-bonded tooth was sectioned into several slices and its adhesion area was trimmed to 1 mm² to form dumbbell-shaped specimens to obtain more and additional information from one tooth⁴. Shono et al.⁵ further modified the microtensile test by sectioning a tooth into rectangular sticks, 1×1 mm in cross-section, without trimming after
sectioning, and referred to it as the non-trimming microtensile test. It is expected that this method can supply more detailed analysis within one tooth.

Miyazaki reported that the adhesive failure tended to increase while the cohesive failure in the adhesive layer tended to decrease with decreasing cross-sectional area in the conventional tensile test. However, it remains unclear whether there is any difference in the failure modes between the conventional test and the microtensile test. The stress concentration to cause failure depends first on the external shape in the dumbbell type specimen but on the internal structure in the non-trimming small specimen as well as in the conventional bulk specimen. It is essential not to select a testing method that shows a clear distinctive stress concentration for comparing the failure mode in adhesion testing. The present study, therefore, aimed to compare the failure mode of the conventional tensile test with that of the non-trimming microtensile test using two bonding systems available on the market, to clarify the better method for evaluating the advanced dentin bonding systems.

**MATERIALS AND METHODS**

Freshly extracted caries-free human third molars were provided for this study. Two kinds of dentin bonding systems were selected as listed in Table 1: one self-etching bonding system (code: CLB) and one wet bonding system (code: MSB) and each respective composite resin (Clearfil AP-X, Kuraray, Tokyo, Japan and Z100, 3M, St. Paul, USA). The superficial dentin was exposed by cutting the tooth through the occlusal enamel-dentin junction with a low speed diamond saw (Isomet, Buehler, Lake Bluff, USA) under water-cooling.

In the case of the conventional tensile test, the as-cut dentin was exposed to 5 mm in diameter using masking tape and treated with the dentin bonding system. As seen in Table 1, the CLB and MSB are applied with a self-etching primer and an etchant, respectively, prior to applying bonding agent. All the procedures for bonding were performed according to the manufacturers’ instructions. A plastic tube 5 mm in inner diameter and 5 mm in height was put on the treated dentin and the composite resin was filled into it. The resin was cured by 40-second radiation of visible light two times. The specimen was immersed in water for 24 hours before the test. The conventional tensile test was performed with a universal tensile testing machine (Tensilon-III-500, TOYO Boldwing, Tokyo, Japan) at a crosshead speed of 2 mm/min. Five specimens were provided for each dentin bonding system.

The non-trimming microtensile test specimen was prepared according to the

<table>
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<th>Table 1 Materials used</th>
<th>Batch number</th>
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<tr>
<td></td>
<td>Code</td>
<td>Etchant</td>
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<tr>
<td>Clearfil Liner Bond ⅡΣ</td>
<td>CLB</td>
<td>—</td>
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<tr>
<td>Single Bond</td>
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method reported by Shono et al.5,7). The dentin was treated and the resin was photocured in a similar manner to the preparation of the conventional tensile specimen mentioned above, except for the size of the resin which was approximately 5×5 mm in area and 5 mm in height. After stored in water for 24 hours, the resin-bonded tooth was sectioned along the tooth axis to form rectangular sticks, 1×1 mm in cross-section, as the non-trimming small specimen as shown in Fig. 1. About 15 specimens were obtained from one tooth and three teeth were used for each dentin bonding system. Both sides of the specimens were adhered to the attachments by cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, USA).

According to the preliminary experiment showing no differences in the failure mode between the crosshead speed of 1 and 2 mm/min for both the conventional tensile and non-trimming microtensile tests, the lower speed of 1 mm/min was adopted in the microtensile test because the specimen was small.

After tensile testing, the fracture surface was observed under a scanning electron microscope (S-3000N, Hitachi, Tokyo, Japan) to clarify the failure mode of each specimen. The fracture surfaces were classified into four categories. They were (1) adhesive failure: fracture at the interface between the bonding agent and dentin involving the resin-infiltrated layer, (2) cohesive failure in the bonding agent, (3) cohesive failure in the dentin, and (4) cohesive failure in the composite resin. The identification of the fracture surfaces was performed on the basis of their character-

Fig. 1 Schematic procedure of non-trimming microtensile test6).
istic surface structural appearance by secondary electrons as well as the component image by backscattered electrons with SEM for the antagonistic faces of the fractured specimen. The NIH software was used to measure the area of each fracture surface.

The results of the bond strength as well as the fraction of the fracture surface were analyzed by analysis of variance (ANOVA) and t-test.

RESULTS

The tensile strengths obtained from the conventional tensile test and non-trimming microtensile test are shown in Fig. 2. No significant difference was found in bond strength between CLB and MSB by the conventional tensile test ($p>0.05$). In the non-trimming microtensile test, however, the bond strength with MSB was significantly larger than that with CLB ($p<0.05$). It was also found that the non-trimming microtensile test showed a significantly larger bond strength than the conventional tensile test for each bonding system ($p<0.05$), while in the former the standard deviation was markedly larger.

Fig. 3 shows an example of the secondary electron image (SEI) and back scattered electron image (BEI) exhibiting the component image for a fracture surface after the non-trimming microtensile test.

Figs. 4 and 5 show representative SEM images of the fracture surface by the conventional tensile test for CLB and MSB, respectively. The surface was inclined by 45 degrees to emphasize the surface irregularities. The particular phases were distinguished by referring to the corresponding BEI. Clearly different failure modes were
observed between the center and periphery of the resin-dentin bonded area. The center was widely occupied by the cohesive failure in dentin, while the adhesive failure and the cohesive failures in the resin and bonding agent layers were complicatedly mixed at the periphery.
Fig. 6 Representative fracture surface of a non-trimming microtensile test specimen treated with CLB observed by SEM under 45 degrees inclination. (Original magnification: ×80)
D: cohesive failure in dentin
R: cohesive failure in composite resin
B: cohesive failure in dentin bonding agent
I: adhesive failure at interface

Fig. 7 Representative fracture surface of a non-trimming microtensile test specimen treated with MSB observed by SEM under 45 degrees inclination. (Original magnification: ×80)
D: cohesive failure in dentin
R: cohesive failure in composite resin
B: cohesive failure in dentin bonding agent
I: adhesive failure at interface

Fig. 8 Fraction of failure modes by different testing methods for two dentin bonding systems.
Figs. 6 and 7 also show the representative fracture surfaces of the non-trimming small specimens by the microtensile test. These specimens were extracted from the area near the center where the simple cohesive failure in dentin appeared by the conventional test. Unlike the large specimen, they showed mixed failures composed of larger parts of adhesive failures. Such a failure mode was not markedly different at the periphery of the bonded area.

All the failure modes for different tests and bonding systems are summarized in Fig. 8. The conventional test showed more cohesive failure in dentin and lesser adhesive failure, while significantly increased adhesive failure was obtained by the non-trimming microtensile test for the two bonding systems. The fraction of the adhesive failure in the CLB-treated specimen was especially high at 50%.

DISCUSSION

The present results showed that regardless of the type of the dentin bonding system the average bond strengths obtained by the non-trimming microtensile test were larger than those obtained by the conventional tensile test. Cardoso et al. compared the bond strength among the shear test, tensile test and microtensile test and reported that the mean value of the microtensile test was higher than that of the shear test or tensile test. Sano et al., Shono et al. and Watanabe et al. also demonstrated the larger bond strength in their microtensile tests using dumbbell-shaped small specimen in the former and non-trimming specimens in the latter two, respectively. Sano et al. suggested the reason for the larger bond strength in their test was that the small adhesive interface used in the microtensile test contained fewer defects compared with the larger specimens. This may be true especially when the small specimen is prepared by first cutting the tooth into the specified shape and size prior to the bonding procedures. Since the tooth is sectioned into small specimens after bonding in the usual microtensile test, however, the defects if any in the bulk resin-dentin interface should distribute over the small specimens with the same probability as in the specimen for the conventional test. Thus, the actual small specimens cannot always contain lesser defects as Sano et al. expected.

In the dumbbell-shaped specimens, considerably larger parts of the bonded area are trimmed off at the resin-dentin interface. The non-trimming small specimens, in contrast, are prepared by merely sectioning the resin-bonded tooth, and their total interface except that the cutting allowance should be almost equivalent in the bonding aspects involved in the distribution of the bonding defects to the large specimen in the conventional test. However, the microtensile test using the non-trimming small specimens in the present study showed significantly larger bond strength on average than the conventional tensile test for the two bonding systems. In addition, the standard deviation was also significantly larger in the non-trimming specimens. Some specimens showed a bond strength as large as twice to three times the conventional bulk specimen and some showed nearly 0 MPa. These results appeared to be associated with the failure mode reflecting the heterogeneous bonding throughout the
Pashley et al. reported that the simple shear or tensile bond test served well when resin-dentin bond strength was relatively low; however, as the bonding system improved, the bond strength became high enough to cause cohesive failure in dentin. Especially the application of dentin primer on dentin promoted the formation of dentin-infiltrated resin layer and was confirmed to increase the adhesive strength. If the cohesive failure in dentin is detected by the conventional tensile test, it may sometime lead to a misinterpretation that the improved dentin bonding system possesses a good adhesion performance to dentin.

In the comparison of CLB and MSB in the conventional tensile test in the present study, the adhesive failure occupied 17 and 23% of the whole fracture surface while the cohesive failure in dentin occupied 44 and 30% in the former and the latter, respectively. Judging from the general concept, the CLB exhibiting smaller fraction of adhesive failure and larger cohesive failure in dentin appeared to show a better adhesion performance to dentin than MSB. However, the bond strength with CLB was not larger than that with MSB. This appeared inconsistent with each failure mode as it is natural that the adhesive failure should change to the cohesive failure as the bond strength is improved. As seen in Figs. 4 and 5, the center portion of the large specimen, which should involve some adhesive failures, was evenly covered by the cohesive failure in dentin. It was suggested from the comparative observation that once a crack entered the dentin layer or initiated there in the large specimen it would rapidly pass through the dentin in a manner of a brittle fracture even if there were more defective regions at the interface, so long as they were not predominantly weaker than the dentin.

The non-trimming microtensile test showed quite different fracture surfaces from the conventional test. The adhesive failure was significantly increased and the cohesive failure in dentin was significantly decreased in the small specimens. In CLB the adhesive failure reached as high as 50% of the whole surface. Between the two bonding systems, MSB showed significantly less adhesive failure and two fold more cohesive failures in dentin and resin than CLB. The bond strength was significantly larger in MSB and it was coincident with the tendencies in failure mode. These results suggest that the adhesion performance was significantly improved in MSB than in CLB. It was also suggested that the non-trimming microtensile test might be able to provide more detailed, accurate information of adhesion performance than the conventional tensile test.

Such discrepancies between the conventional tensile test and the non-trimming microtensile test appear to result from the different failure modes in association with the adhesion characteristics and the size of the specimen. The failure consists of the initiation and successive propagation of cracks. The crack will initiate at a point such as a notch, defect or weak structure where the stress easily concentrates and then propagate through the weaker region. In the dumbbell-shaped small specimen, the width is deeply reduced at the resin-dentin interface and the fracture is intentionally caused there. It may be favorable to examine the bond strength or
bonding features at a selected site of interface. On the other hand, the non-trimming small specimen has a uniform cross-section along its long axis to avoid notch effects and it cannot be estimated before testing where it may fail. Due to its small cross-section, the specimen will be directly affected by the existence of any defect or mechanically weak structure in the microtensile test. This might be the reason for the large standard deviation of the bond strength in the non-trimming small specimens in the present study. It is assumed that the small specimens in which the interface happened to encounter the defective or deficient adhesion might fail in the mode of adhesive failure with smaller bond strength while the sound small specimen might show a significantly greater bond strength. This is one of the important features of the non-trimming microtensile test, whereas in the conventional test the failure mode of the specimen may be determined by the preferential development of some major cracks but not always by the distribution of the defective or week region.

Thus, the non-trimming small specimens may potentially provide the inherent information on bonding at each sectioned site of interest without being affected by the cracks developed from other defective regions, showing more realistic aspects of resin-dentin bonding than the conventional bulk specimen. In the categories of the failure mode, the failure within the resin-infiltrated dentin layer was involved in the adhesive failure in this study because it was difficult to clearly identify that layer. The need for detection of that layer remains.

The location of the bond to be examined has so far been limited to the center of the occlusal or labial surface of midcoronal dentin in the conventional tensile test\(^1\) because of its experimental conditions. With the non-trimming microtensile test, any portion will be available for specimens. However, the microtensile test cannot be an alternative to the conventional tensile test and it is emphasized that the selection of the testing method should depend on the purpose of the test. The conventional test is adequate to evaluate the practical significance of the composite resin restoration in terms of the macroscopic bond strength. More detailed analysis of bonding aspects utilizing the foregoing advantages of the non-trimming microtensile test will be useful for further improvement of the bonding systems, although it requires time and needs special attention to prepare the specimens.

REFERENCES


