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Fractography of interface after microtensile bond strength test using swept-source optical coherence tomography

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ABSTRACT

Objective. To determine the effect of crosshead speed and placement technique on interfacial crack formation in microtensile bond strength (MTBS) test using swept-source optical coherence tomography (SS-OCT).

Materials and methods. MTBS test beams ($0.9 \times 0.9\,mm^2$) were prepared from flat human dentin disks bonded with self-etch adhesive (Clearfil SE Bond, Kuraray) and universal composite (Clearfil AP-X, Kuraray) with or without flowable composite lining (Estelite Flow Quick, Tokuyama). Each beam was scanned under SS-OCT (Santec, Japan) at 1319 nm center wavelength before MTBS test was performed at crosshead speed of either 1 or 10 mm/min ($n = 10$). The beams were scanned by SS-OCT again to detect and measure cracks at the debonded interface using digital image analysis software. Representative beams were observed under confocal laser scanning microscope to confirm the fractography findings.

Results. Two-way ANOVA showed that for MTBS the crosshead speed was not a significant factor ($p > 0.05$), while there was a difference between placement techniques ($p < 0.001$), with flowable lining yielding higher mean values. On the other hand, for crack formation, there was a significant difference between crosshead speeds ($p < 0.01$), while the placement technique did not show up as a statistically significant factor ($p > 0.05$). The interaction of factors were not significant ($p > 0.05$).

Significance. Testing MTBS samples at higher crosshead speeds induced more cracks in dentin. Lining with a flowable composite improved the bonding quality and increased the bond strength. SS-OCT can visualize interfacial cracks after restoration debonding.

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1. Introduction

Despite the widespread utilization of resin composite to recover function and esthetics of teeth, creating interfacial gap-free restorations remains a challenge, which may lead to adhesion failure between resin and substrate [1,2]. Therefore, lining technique with low-viscosity composite at the floor combined with the self-etch bonding system is recommended to improve the interfacial adaptation and sealing [3–5].

The conventional in vitro tests to assess the performance of restorative materials and their associated placement techniques encompass marginal integrity and bond strength evaluation. Microtensile bond strength (MTBS) test permits the determination of regional bond strength of a small area in the bonded complex substrates, and has proved to be reliable for screening the effectiveness of adhesives [6,7]. Despite the similarity of the employed materials and testing procedures, the results among studies have shown diversity to some extent. Some parameters to consider in bond strength test related to the specimen design and the test mechanics include the fixation mode, preparation, modulus of elasticity, geometry of specimen, loading configuration and the crosshead speed. The wide difference in bond strength estimation and the inadequacy of standardized laboratory protocols may have expedited the equivocation in interpretation [8,9].

The commonly utilized speeds for dentin bond strength assessment in both tensile and shear modalities are 0.5, 1.0, 2.0 and 5.0 mm/min. Although it was reported that bond strength values were not significantly different between experimental groups using speeds ranging from 0.01 to 10.0 mm/min [9–11], time-dependent strain response in viscoelastic materials might affect the test results at various crosshead speeds [10]. Besides, the chewing cycle and jaw closing movement of human range around 800 ms and 400 ms, respectively [12], corresponding to a speed of approximately 2000 mm/s, which is 500 times superior to what may be usually used in bond strength testing [13]. The impact of increased crosshead speed on dentin in terms of structural damage is not fully understood. This could be partly due to the limitations of conventional in vitro microscopic imaging methods, making such evaluation challenging. In this regard, non-invasive detection of defects developing due to load in dentin would be of importance.

Optical coherence tomography (OCT) is a real-time, high resolution optical imaging modality based on the backscattered signal intensity from within structure [14]. Its applications in dentistry include assessing dental caries [15], monitoring defects of restoration [16–21], and determining tooth crack locations [22,23]. Few studies to date have utilized this tool for exploration of interfaces in bond strength test specimens.

Therefore, the aim of this study was to determine the effect of crosshead speed on the bonding interface with and without lining flowable composite using OCT and confirm the findings by cross sectional confocal laser scanning microscopy (CLSM). The null hypotheses were that: there was no effect of crosshead speed and placement technique on (1) MTBS and (2) interfacial crack formation after MTBS test.

2. Materials and methods

2.1. OCT system

A swept-source OCT system (IVS-2000, Santec, Komaki, Japan) (SS-OCT) was used in this study with the spectral bandwidth of the laser centered at 1319 nm at a 20 kHz sweep rate. The probe power of 5 mW does not exceed the safety limits defined by American National Standard Institute (ANSI) [24]. The lateral resolution of 20 μm is determined by the objective lens at the probe. The focused beam scans the item of interest in two-dimensions X and Y. The axial resolution of the system is 12 μm in air, corresponding to 7 μm in tissue assuming a refractive index of approximately 1.5 [25]. The sensitivity of this system is 106 dB and the shot-noise limited sensitivity is 119 dB. Backscattered light carrying information about the microstructure of the sample is collected, returned to the system, digitized in time scale and then analyzed in the Fourier domain to reveal the depth information of the subject. The analysis of the frequency components of backscattered light from the specimen produces reflectivity profile called A-scan. Serial A-scans generate 2-D cross-sectional B-scan from which a high-resolution gray-scale image can be obtained.

2.2. Specimen preparation

In this study, 24 extracted sound, unrestored human premolars were collected according to the patients' informed consent, as approved by the Institutional Review Board of Tokyo Medical and Dental University, Human Research Ethics Committee, protocol no. 725 and stored frozen (−20 °C). After cleaning with a dental scaler and removing the root, buccal surfaces of the teeth were ground with a model trimmer (Y-230; Yoshida, Tokyo, Japan) to expose dentin and wet-polished with 600-grit silicone carbide (SiC) paper (Sankyo, Saitama, Japan). The restoration materials in this study are listed in Table 1. Self-etching primer agent of Clearfil SE Bond (Kuraray Noritake Dental, Tokyo, Japan) was applied to the entire dentin surface with a disposable brush tip for 20 s and gently air blown, then coated by a layer of adhesive, followed by mild airflow and curing with a halogen light curing unit (Optilux 501, Kerr, CA, USA; 600 mW/cm^2 intensity). A flowable composite (Estelite Flow Quick, Tokuyama Dental, Japan) was applied as a thin liner in half of the specimens followed by an increment of universal composite of Clearfil AP-X (Shade A2, Kuraray Noritake Dental, Tokyo, Japan). In the remaining specimens, a single increment of the universal resin composite was placed on top of the adhesive layer without lining. Each composite layer was cured separately for 20 s using the light curing unit. SS-OCT was used to monitor the procedure of dentin exposure for total enamel elimination, application of flowable composite for its uniform thickness, and restoration of the universal composite for interfacial adaptation. The flowable composite layer measured under OCT was at 0.5 mm in optical thickness, equal to approximately 0.3 mm in real thickness. After storage in water at 37 °C for 24 h, the specimens were sectioned using a water-cooled slow speed diamond saw (Isomet, Buehler, IL, USA) into 0.9 mm × 0.9 mm sticks with their long axis perpendicular to the bonding interface. Each