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Determining the temporal development of dentin-composite bond strength during curing

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ABSTRACT

Objectives. As composite restorations cure a competition develops between bond formation and shrinkage stress at the composite-dentin interface. Thus, understanding the temporal development of tooth-composite bond strength should enable better assessment of tooth-composite debonding.

Methods. In this study, bond strengths of composite-dentin specimens obtained from tensile test at different curing times were used to determine the bond formation rate. By varying the composite thickness and output from the curing light, their effects on the rate of bond formation for two different materials (a conventional and a bulk-fill composite) were also investigated. The proportions of cohesive and adhesive failure were determined by analysis of electron micrographs of the fractured surfaces.

Results. The development of dentin-composite bond strength (S) with time (t) can be described by the equation: $S = S_{max}(1 - \exp(-\alpha t))$, where S_{max} is the final bond strength (~12 MPa for both composites) and α the rate of bond formation. Using bulk-fill and thinner specimens gave faster bond formation. In fact, the higher the irradiance at the interface, the higher the rate of bond formation. However, α had a maximum value of ~0.6 s^{-1} and the rule of reciprocity did not hold. A minimum dose of ~2 J/cm^2 was required to achieve adequate bond strength. The predominant failure mode changed from cohesive in the composite and adhesive to interfacial at the adhesive-dentin interface, indicating the latter to be the weakest link in the cured dentin-composite assemblies considered.

Significance. When combined with the temporal development of shrinkage stress, the current results will help determine the likelihood of tooth-composite debonding.

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1. Introduction

Since their first introduction to restorative dentistry in the late 1950s, light-cured resin-based composites (RBCs) have undergone many improvements to enhance their mechanical properties and esthetics [1]. These developments have made RBCs the most widely used dental materials for the repair of dental caries, crown fractures, and tooth wear. However, polymerization shrinkage, which can lead to marginal gaps and, ultimately, failure of the restoration through the development of secondary caries, remains a major drawback of RBCs [2]. During polymerization, the methacrylate double bonds of the resin re-hybridized and link together to form polymeric networks resulting in volumetric shrinkage as the molecular distances and free volume are reduced [3].

The shrinkage kinetics of dental composites during light curing has been studied extensively using dilatometry [4], the bonded-disc method [5] and digital image correlation (DIC) [6,7]. The results show that the majority of shrinkage in light-cured composite resins occurs during the first few seconds of the polymerization process [7]. Clinically, composite shrinkage is restricted by adhesion to the cavity walls, which results in the development of internal stresses [8]. The latter are a product of the polymerization shrinkage, the constraints imposed by the surrounding tooth and the rigid nature of the reinforced cross-linked polymer network formed during curing [3]. The shrinkage stress produced in thin disk specimens also shows rapid increase at the very beginning [9].

In conjunction with the development of RBCs, adhesive technology has evolved considerably during the last 50 years, leading to much improved tooth-composite bond strength [10,11], bonding procedures [12] and antibacterial capabilities of adhesive compositions [13]. For self-etch systems, the micro-tensile bond strength can reach 30 MPa [11]; whereas 40 MPa has been reported for etch-and-rinse systems [10]. However, debonding of composite restorations still occurs clinically [14,15]. Some studies further show that debonding can happen during the curing process [16,17]. It should be recognized that, like shrinkage strain and stress, bonding between the tooth and composite also takes time to form, and the rate of formation among these entities could be different. There is, thus, a competition at the composite-tooth interface between bond formation and development of shrinkage stress. Even though the final bond strength may be higher than the final shrinkage stress, it is entirely possible that debonding can still happen when, for example, shrinkage stress is developing much faster than the tooth-composite bond. To avoid debonding, therefore, the developing bond strength must be higher than the developing shrinkage stress at all times. Studies on dental adhesion almost exclusively focus on measuring the final value of bond strength. To date, only Davidson et al. [18] have investigated the bond strength development of a chemically cured micro-filled composite, which showed a gradual increase over a period of 30 min. They also investigated the small increase in bond strength of a light-activated composite over a period of 30 min post-curing. For a light-cured resin composite during curing, the increase will be much faster and its measurement more challenging. This probably explains the lack of studies on the temporal bond strength development of composite during its light curing.

The objective of this study is to determine the time-dependent formation of the dentin-composite bond by measuring the bond strength at different time points during light curing. The effects of the composite's thickness and the intensity of the curing light on the rate of bond strength development will also be investigated for a conventional and a bulk-fill composite, in response to the latter's introduction for deep cavities in recent years.

2. Materials and methods

2.1. Preparation of the specimens

Bovine incisors that had been cleaned and stored in distilled water at 4 °C for 2 weeks were used for preparing the specimens. Five tooth slices of about 3.5-mm thick were obtained from each bovine incisor (Fig. 1a and b) by cutting the teeth perpendicular to the long axis using an Isomet™ low-speed diamond saw (Buehler, Lake Bluff, IL, USA). After removing the residual pulp tissue, a high-speed handpiece with a diamond bur was used to cut the tooth slices into two halves (Fig. 1c). These were then trimmed into dentin slabs (Fig. 1d) along the dashed lines shown in Fig. 1c using the high-speed handpiece. The final dimensions of the dentin slabs were: width 3 mm × length 4 mm × height 2 mm (Fig. 1e). A total of 540 dentin slabs were prepared as described. Two commercial materials, a conventional composite (Filtek™ Z250, 3M ESPE, St. Paul, USA) and a bulk-fill composite (Filtek™ Bulk Fill, 3M ESPE, St. Paul, USA), both shade A2, were used in this study. The composition and other product information of these composites are listed in Table 1. In order to investigate the effect of composite thickness on the rate of bond formation, blocks of cured composite (length 11 mm × width 8 mm) with three different thicknesses (1, 3 and 4 mm) were fabricated using the Teflon molds shown in Fig. 1f, g and h. When filled with one of the composite materials, the molds were pressed with a glass slide, and the composite cured for 20 s using a LED curing unit (Elipar™ S10, 3M ESPE, St. Paul, USA) with an irradiance of 1200 mW/cm^2. The three subgroups generated for each material were: Z1, Z3 and Z4 for Z250, and B1, B3 and B4 for Bulk Fill, representing 1-mm, 3-mm and 4-mm thick specimens, respectively. There were 45 blocks in each subgroup, giving a total of 270 blocks.

2.2. Bonding procedure

Before assembling a dentin-composite specimen, a layer of adhesive resin was pre-cured on both the inner (next to the root canal) surface of the dentin slab and the upper surface of composite block. To standardize the procedure, dentin surfaces were polished with a 600-grid SiC paper (Buehler, Lake Bluff, IL, USA) under water irrigation for 30 s. The exposed dentin surfaces were etched with 35 percent phosphoric acid (Scotchbond Etchant, 3M ESPE) for 15 s, rinsed for 15 s using distilled water and blot-dried for 2 s. Immediately after blot drying, two consecutive coats of adhesive (Adper™ Single Bond Plus, 3M ESPE, St. Paul, USA) were applied and agitated