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Relationship between thin-film bond strength as measured by a scratch test, and indentation hardness for bonding agents

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ABSTRACT

Objectives. To evaluate thin-film bond strength between a bonding agent and human dentin, using a scratch test, and the characteristics and accuracy of measurement.

Methods. One-step bonding agents (BeautiBond; Bond Force; Adper Easy Bond; Clearfil tri-S Bond) and two-step bonding agents (Cleafil SE Bond; FL-Bond II) were investigated in this study. Flat dentin surfaces were prepared for extracted human molars. The dentin surfaces were ground and bonding agents were applied and light cured. The thin-film bond strength test of the specimens was evaluated by the critical load at which the coated bonding agent failed and dentin appeared. The scratch mark sections were then observed under a scanning electron microscope. Indentation hardness was evaluated by the variation in depth under an applied load of 10 gf. Data were compared by one-way ANOVA with the Scheffé's post hoc multiple comparison test ($p < 0.05$). In addition, thin-film bond strength and indentation hardness were analyzed using analysis of correlation and covariance.

Results. The thin-film bond strength of two-step bonding agents were found to be significantly higher than that of one-step bonding agents with small standard deviations. Scratch marks consistently showed adhesive failure in the vicinity of the bonding agent/dentin interface. The indentation hardness showed a trend that two-step bonding agents have greater hardness than one-step bonding agents. A moderately significant correlation ($r^2 = 0.31$) was found between thin-film bond strength and indentation hardness.

Significance. Thin-film bond strength test is a valid and reliable means of evaluating bond strength in the vicinity of the adhesive interface and is more accurate than other methods currently in use. Further, the thin-film bond strength is influenced by the hardness of the cued bonding agent.

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1. Introduction

Many studies have been conducted to examine the bond strength of dentin bonding agents using shear and tensile bond strength tests [1–4] by fracturing resin composite, bonding agent and tooth. However, the fracture patterns in these tests show more than one failure mode, such as adhesive failure between the bonding agent and dentin, cohesive failure within the dentin, cohesive failure within the resin composite, and mixed failure modes [1,2,4]. Therefore, such bond strength tests do not solely reflect adhesive failure between the bonding agent and dentin.

In this study, the scratch test was used to evaluate the adhesive strength of bonding agents to human dentin. In the scratch test (thin-film bond strength test) an indenter is drawn across the coated surface under incremental and progressive loading to generate a scratch—damage or delamination. In industrial circles this test is used as a measure of adhesive strength [5–7]. For example, for cutting tool coatings, such as TiC or TiN [6]; coating adherence [7] for diamond-like carbon (DLC) protective coatings for computer hard discs; and evaluation of adhesion/cohesion bond strength of the plasma spray coatings [8]. In dentistry, this test was reported for the evaluation of an adherent apatite coating on a titanium substrate [9], to investigate wear resistance and wear mechanisms of dental composite resins [10,11], and to determine the hardness and amount of chipping for dental ceramics [12]. However, no reports have been found for adhesion evaluation, although mechanical properties were measured for bonding agents [13].

We previously reported that the thin-film bond strength test correlates with the tensile bond strength test [14]. The thin-film bond strength of dentin bonding agents is characterized by a critical failure load. Therefore, the relationship between thin-film bond strength by the scratch test, and indentation hardness for bonding agents, was evaluated.

2. Materials and methods

2.1. *Adhesive systems used in this study*

As shown in Table 1, four one-step and two two-step bonding agents were investigated in this study.

2.2. *Thin-film bond strength test*

2.2.1. *Specimen preparation*

Extracted human molars were used and flat dentin surfaces were prepared with the coronal part of the tooth using a low-speed diamond saw (Isomet, Buehler Ltd; Lake Bluff, USA). The dentin surfaces were ground on wet 600-grit silicon carbide paper to prepare a flat dentin surface. Bonding agents were applied to the dentin surface and light cured according to manufacturer's instructions (Table 1). A curing light was used Dent Craft Blue Lex (Yoshida; Tokyo, Japan) with a power density of >900 mW/cm^2. Specimens were stored in distilled water at 37 °C for 24 h. The use of human teeth was approved by the Ethics Committee of Asahi University (No. 23112).

2.2.2. *Thin-film bond strength measurement*

The scratch test of the bonding agents to dentin was measured 10 times for each bonding agent at a sample sliding speed of 5.25 mm/min, and variable loads of 0.03–15.0 N using a 200 μm radius diamond Rockwell indenter. This produced a scratch approximately 7 mm in length, using a Micro Scratch Tester (CSEM Instruments; Peseux, Switzerland). The thin-film bond strength was evaluated by measuring the critical load at which the coated bonding agent failed and the dentin surface began to appear. The thin-film bond strength test is measured by an indenter sliding over the coated surface under an incremental and progressive load, and generating damage or delamination (Fig. 1).

2.2.3. *SEM observations*

After the thin-film bond strength test, the same scratch marks of delaminating were cross-sectioned perpendicular to the scratched surface, using a low-speed diamond saw. The scratch mark sections were observed under a scanning electron microscope (SEM).

Scratch mark sections were prepared as follows: (i)–(iii)

i) Not ground.
ii) Ground with wet 600- up to 1500-grit silicon carbide paper and 6 down to 0.25 μm diamond paste.
iii) After (ii), immersed in 6N HCl for 40 s, rinsed with distilled water, and then soaked in 1 wt% NaOCl for 60 min.

The specimens were chemically prefixed with 2.5% glutaraldehyde solution (4 °C; 2 h) and rinsed twice with cacodylate buffer (4 °C; 20 min × 2). They were then chemically fixed with 1% OsO_4 solution (4 °C; 1 h) and rinsed twice with cacodylate buffer (4 °C; 20 min × 2). The specimens were then dehydrated in a graded series of ethanol, immersed in t-butanol (20 min × 2), and freeze-dried (VFD-21S^{TM}; Vacuum Device, Ibaragi, Japan). Finally, the samples were placed on aluminum stubs, coated with osmium for 10 s (HPC-1S^{TM}; Vacuum Device, Ibaragi, Japan), and observed by SEM (S-4500^{TM}; Hitachi, Tokyo, Japan) at 300 and 3000× magnification.

2.3. *Indentation hardness test*

Bonding agents were placed in disc-shaped molds (10 mm diameter, 7-mm thick) between two glass slides and light cured for 60 s on each side. However one-step bonding agents were evaporated by air blowing and light cured because water, ethanol, and acetone were included as solvents, and inhibit polymerization. After storage in air at 37 °C for 24 h, the bonding agent surfaces were ground flat with wet 600-grit silicon carbide paper to achieve a 5 mm thickness. In addition, human dentin (5 mm thick) specimens were ground and used as controls. Indentation hardness was evaluated with a Vickers indenter with a Dynamic Ultra-micro Hardness Tester (DUH-200, SHIMADZU Corp. Kyoto, Japan) and measured 10 times for each specimen. A load of 10 gf was applied to the specimens for 15 s (Fig. 2). Fig. 3 shows the variation in indentation depth under an applied load of 10 gf, and the formula for calculating indentation hardness.