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Aspects of adhesion tests on resin–glass ceramic bonding

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ABSTRACT

Objectives. This study aimed to compare and contrast two resin–ceramic bond strength tests, the tensile bond strength and the four-point bending tests. The effects of hydrofluoric acid (HF) etching time and storage condition on bond strength were also studied.

Methods. Ceramic beams (N = 480) with the dimensions of $2.00 \times 2.00 \times 12.45\,mm^3$ were sectioned from lithium disilicate ceramic ingots (IPS e.max CAD), then polished and fired for final crystallization. The joint surfaces were etched with HF gel (IPS Ceramic etching gel) for 20 s, 40 s, or 60 s of each group (n = 160). Then, a silane coupling agent (Vitasil®) was applied in a single application on the HF etched surfaces, left for 60 s before air-drying. Two beams were bonded together with resin composite cement (Variolink II®) in a tailored-mold ($2.00 \times 2.00 \times 25.00\,mm^3$) to control cement thickness to 0.10 mm and then light cured on both sides. The bonded specimens were further divided into two groups (n = 40): (1) tested one day after luting (dry); and (2) tested after storage in 37° C distilled water for 4 weeks. Two mechanical tests were used (n = 20): the tensile bond strength and four-point bending tests. Bond strength results were subjected to two-way AoV, and Weibull statistics with $\alpha = 0.05$. Fracture surfaces were examined visually and verified using light microscopy.

Results. The four-point bending test showed a higher consistency than the tensile bond strength test using Weibull statistics ($p < 0.01$). The effect of HF etching time on the flexural strength was significant, with longer HF etching times decreasing the flexural strength ($p < 0.01$). Storage also had a significant effect on the flexural strength ($p < 0.01$). However, HF etching time did not have a significant effect on the tensile bond strengths ($p > 0.05$) and the influence of the storage time was marginally significant ($p < 0.05$). More than 75% of specimens failed adhesively in the four-point bend test while a mixture of adhesive, cohesive and mixed failures was observed in the tensile bond test.

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Significance. The four-point bending test might be a better approach to evaluate bond strengths. Increased HF etching time and a longer storage period resulted in a decrease in the flexural bond strength. However, both HF etching time and storage time had no significant effect on the tensile bond strengths.

1. Introduction

It is unquestionable that ceramic has become widely employed in dental restorations as its superior mechanical properties and better appearance have improved substantially [1,2]. However, there remains a major constraint for clinical application of ceramic materials, they are vulnerable when subjected to tensile stresses [3], *i.e.* ceramics are prone to fracture that is a failure. Despite most restoration failure originating from ceramic fracture, the bonding reliability of the ceramic is a possible explanation for restoration failure and should not be overlooked.

Other than the performance of ceramic or resin cement, adhesion to a ceramic material is the one of the key factors to evaluate the bond durability (adhesion strength)[4], *i.e.,* long term success in clinical application for an adhesion system of an indirect restoration [2]. Current procedures for tooth preparation now aim to preserve as much dental hard tissue as possible [5], hence, restoration retention mainly relies on adhesion to the prepared tooth [3]. Therefore, adhesion is crucial and should be thoroughly evaluated in order to examine and understand bond durability.

To begin with, there are adhesive interfaces. In a traditional indirect restoration adhesive system, there are always two adhesive interfaces: ceramic to resin cement and tooth structure to resin cement [3]. Various studies have evaluated the ceramic to resin [4] and tooth to resin [6,7] bonds in order to better understand and enhance the adhesive strength [8]. Other than the adhesive interface, the bonding mechanism has also been studied extensively. There are two mechanisms involved in ceramic to resin cement bonding, namely, micromechanical interlocking and chemical bonding [2]. To create a micromechanical interlock, hydrofluoric acid etching [3,4,9] or, sometimes, grit-blasting [10–12] of the ceramic surface are the usual methods. Application of a silane coupling agent on etched glass ceramic surface is mandatory to create durable chemical bonding [6,8,13–16].

As mentioned, to determine whether a restoration is successful, not only needs the individual performance of ceramic and resin cement to be considered, but also the actual bonding mechanisms between the ceramic and resin cement must also be studied. To evaluate durable bonding in a laboratory setting, various bond strength tests have been developed [17,18]. The strength to hold the adherend components together is denoted as the bond strength (adhesion strength). A typical model for bond strength testing involves either a (pre-)treated tooth or ceramic specimen joined to a resin composite block (specimen) with resin-based luting cement [3]. The bond strength is calculated by dividing the maximum load to break the bonded specimen by the actual bonding area [2]. Bond strength tests can be categorized into two main types: tensile [3,7,19–25] and shear [26–29], depending on the primary stress applied to the interface. Nevertheless, bearing in mind that there is no genuine shear strength test in existence in dentistry [17,30].

Debate on bond strength tests has continued, with some researchers challenging the validity of these tests with strong criticism to the experimental methodologies [31,32]. Numerous studies have been performed, not only to evaluate the bond strength, but also to verify the test methods [32]. Among the various laboratory bond strength test methods, the microtensile bond strength (MTBS, μTBS) test is the most popular technique to test ceramic to resin cement bonding [3]. Before the microtensile bond strength test was developed [18], the tensile bond strength (TBS) test was available. The only difference between the micro- and tensile bond strength test is the bonding area, otherwise the test conditions are almost identical. Indeed, for the microtensile bond strength test, which was first proposed by Sano et al. in 1994, the bonding area was set below 1.0 mm^2 so that stress distribution across the bonded interface was suggested to be distributed more evenly [18]. It was also suggested that compared with the traditional tensile bond strength test method, the microtensile bond strength test results in more adhesive failures, *i.e.,* adhesive failures may reveal the ‘true’ bond strength. On the other hand, the microshear bond strength test is the most recently accepted method to test tooth to resin composite cement adhesion [6], *e.g.,* a resin composite component joined to a pre-treated tooth surface. Nevertheless, it seems that the word “micro” is a matter of absolutely arbitrary terminology for the sake of appearance only; it has no real meaning!

Four-point bending test indeed is one of the newest methods to access bond strength, such that only few studies were published to date [33–35]. Although interfacial tension test has been advocated for measuring bond strength, the stress distribution at interface is indeed complex and the specimen preparation as well as the alignment is not as simple as that of the other methods [33,36]. More importantly, four-point bending test has the maximum tensile stress on the convex surface [33] and removed the stress concentration at the surface of adhesive [30], which deemed to be more clinical relevant than direct tension test. However, different from the tensile bond strength test, the four-point bend test requires the specimen to be placed horizontally with the adhesive joint placed centrally where the stress is placed at the adhesive joint and the specimen supported at both ends using a fixed distance. The load leads to bending of the specimen and creates a combination of stresses, namely tension and compression, and therefore, interpretation can be more difficult. At the same time, it is achievable to have most jointed specimens fail