# A Microshear Test to Measure the Bond Between Dental Composites and Dental Substrates

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**Introduction:** In order to determine the real efficacy and durability of polymeric dental adhesives, thorough clinical testing is necessary. However, in practice, long-term clinical testing is expensive and commercial adhesive materials are constantly undergoing change so that the clinical testing necessary to evaluate their long-term properties and success rates (typically four years) becomes impractical. Thus, there is the need for reliable laboratory tests to serve as screening tools to identify promising new dental adhesive systems and improvements to existing systems. Adding to the challenge is the very complex conditions that a tooth cavity presents to achieving effective adhesion of the restorative material to this heterogeneous mineralized substrate. The mineralized tissues of the tooth, enamel with its prismatic rod-like apatitic structure and dentin with its array of dentin tubules, are both anisotropic materials that present variable substrates for adhesive systems. Depending upon the orientation of the rods and tubules with respect to the adhesive interface, variations in bond strength would be expected. The polymeric adhesive restorative, a viscoelastic material, is constantly exposed to a hostile, multi-challenging milieu comprising masticatory stresses, aqueous fluids that can initiate chemical attack, and cyclic exposure to heat and cold. This list of factors that can affect adhesion to tooth structure is not meant to be exhaustive but rather illustrates the complex nature of properly assessing the bond between the restorative and the mineralized tissue, and illustrates the challenge involved in designing a reliable screening test.

Shear bond testing to assess the bond strength between polymeric adhesives and mineralized tissue is widely used due to the ease of specimen preparation and testing. We have designed and developed a new shear test protocol that minimizes the tensile forces at the interface and maximizes the shear forces. Because our bonded surfaces are very small (~0.4 $mm^2$), we are able to test many specimens on a single surface of enamel or dentin, thereby aiding in the conservation of teeth. Additionally, we can perform regional mapping of the tooth structure and can reach temperature equilibrium conditions rapidly, which permits accelerated durability studies.

In the case of enamel, adhesion occurs primarily by micro-mechanical interlocking resulting from the penetration of the unpolymerized resin into a superficial porous zone created by chemical etching (acid or through chelation), forming upon polymerization polymeric tags into the conditioned apatitic structure. Because of the structural anisotropy of enamel, it is thought that variation in enamel bonding sites might influence the bonding strength of direct composite restorative systems to this substrate. The purpose of this study was to study how regional variation in enamel tooth structure and the effect of enamel rod orientation affects the bonding ability of a non-priming adhesive system that employs aqueous phosphoric acid as the etchant. This paper presents some of the opportunities that the microshear bond strength test offers in enhancing our understanding of dental adhesion and in designing dental adhesive systems with enhanced strength and durability.

**Materials and Methods*:** The most important elements of the microshear test come with the design of the test. As seen in the figure, cylinders of cured composite resin are bonded onto a prepared substrate, shear tested at a specified rate, and the interfacial properties calculated. Tooth slices were taken from extracted human molars with the selected enamel region sectioned transversely, obliquely, and parallel (longitudinal) to the enamel prism. Each slice was approximately 1.0 mm thick. The enamel slices were bonded to aluminum tabs by first air abrading the aluminum with 50 $\mu$m aluminum oxide powder followed by application of a cyanoacrylate adhesive. The exposed tooth surface was then resurfaced under water with 320 grit SiC paper. A phosphoric acid gel (K-etchant gel, Kuraray) was then applied to the resurfaced enamel for 30 s and then the surface was rinsed with water and dried but not desiccated, then the Photo-Bond adhesive resin was applied. Subsequently, an iris was cut from a micro-bore Tygon tube that had an internal diameter of approximately 0.7 mm and affixed to the enamel surface, and then the resin composite was injected into the iris. The resin was photo-irradiated with visible light (maximum absorbance peak 470 nm) for 60 s. After curing, the iris was removed and the specimens were stored in distilled water at 37 $^{o}$C for 24 h. The cylindrical specimens were tested in shear at a rate of 0.5 mm/min. The change in load as a function of time was recorded and the shear strength was calculated from the equation $\tau = F/(\pi r^2)$, where $\tau$ is the estimated interfacial shear strength, F is the load at failure, and r is the radius of the resin cylinder.

**Results and Discussion:** Seven specimens were tested for the specimens with rods that were sectioned transversely; the mean shear strength and standard deviation was $(31.12 \pm 3.38)$ MPa. The result for five specimens that were sectioned obliquely was $(27.78 \pm 2.17)$ MPa. The result for eight specimens that were cut parallel to or longitudinally along the prisms was $(13.90 \pm 3.11)$ MPa. The results of this particular study showed that the surface parallel to the enamel prism rods was significantly weaker than were the surfaces cut transversely or obliquely. (One-way analysis of variance, $F = 60.8$; df = 5, 34; $p < 0.05$).

**Conclusions:** A microshear test has been developed that allows one to study adhesion of very small areas (0.4 mm$^2$ to 1.0 mm$^2$) that are not accessible to conventional macroshear testing. Thus, the researcher can now horizontally and vertically map interfacial adhesion of different areas and depths of tooth structure, respectively. Furthermore, this method requires significantly fewer extracted teeth for adhesion studies compared to the typical macro adhesion studies.

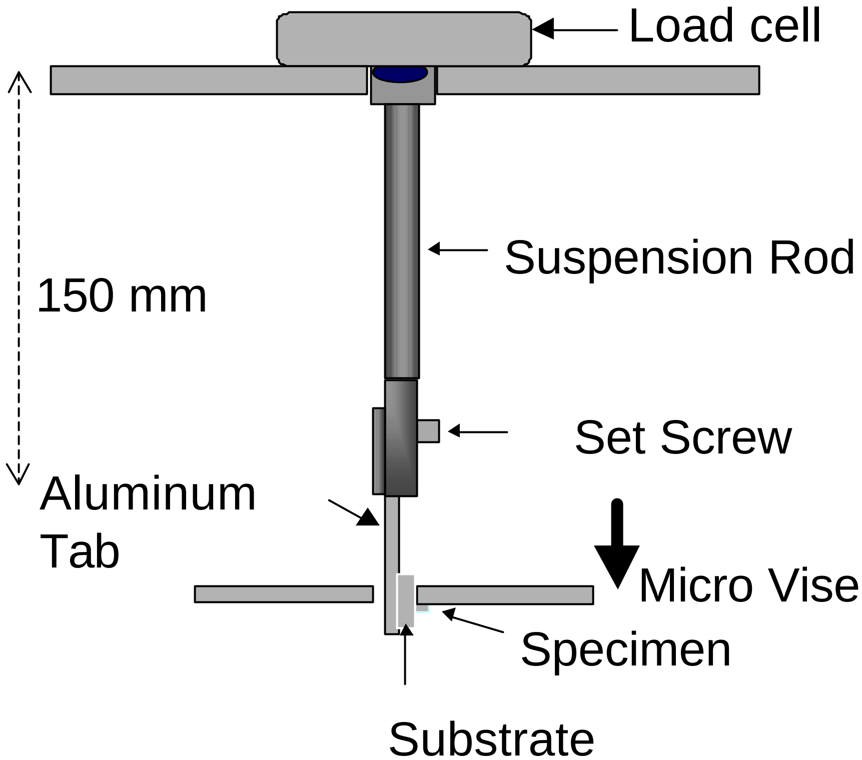


Figure 1: Schematic of the microshear test.

*Materials and equipment are identified only for describing experimental procedure and do not imply recommendation or endorsement.

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