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Method for Immediate Measurement of *In Vitro* Bond Strength of Bonded Direct Esthetic Restorations

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Abstract

There are many different ways to measure the bond strength of direct esthetic restorations to various dental substrates. Unfortunately, most methods cannot measure bond strengths immediately after a restoration has been placed. This lack of clinically-relevant information seriously affects the clinician's ability to select and use various bonding agents and procedures. The aim of this article is to provide a very detailed method for immediate measurement of *in vitro* bond strengths of direct bonded esthetic restorations. It focuses on the steps that should be taken to select and prepare various tooth substrates for bond strength testing, the steps to "restore" various tooth substrates, and to measure the immediate *in vitro* bond strength. A fundamental understanding of a standardized testing protocol should provide clinicians with a clearer appreciation of bond strengths associated with various bonding procedures.

Keywords: Bonding, esthetics, immediate measurement, *in vitro*, enamel bonding, cementum bonding, dentin bonding

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Introduction

It is not uncommon for clinicians to forget the use of "curing" lights with bonded direct esthetic restorations only initiates a chemical process that takes infinite time to complete.[1,2] As a consequence, the bond strengths of bonded direct esthetic restorations to tooth substrates that can be achieved will vary over time as the chemical process continues.[1,2]

There are a number of different methods to measure the bond strength of bonded direct esthetic restorations to various substrates using a variety of bonding procedures. Unfortunately because of preparation limitations, most results are reported as 1-hour, 24-hour, or even longer bond strengths.[3,4,5,7] While the results are reproducible, they do not indicate what may be expected immediately after placement of bonded direct esthetic restorations when shrinkage stresses are developing, when finishing and polishing procedures are performed, or when occlusal adjustments must be made.[5,6]

This article presents a step-by-step description of a method for immediate measurement of *in vitro* bond strength of bonded direct esthetic restorations. The method may be adapted to examine a variety of effects produced by different substrates, substrate preparations, bonding application procedures, and long-term thermal cycling and staining. The method may also be used for studies of bonded indirect esthetic restorations.

Tooth Selection and Preparation

Careful tooth selection and preparation for a variety of different types of studies involving *in vitro* bond strength of bonded esthetic restorations is essential for a reliable outcome. The steps in that process are described below:

Step 1: Determine the Proper Sample Size

Generally, collecting a sufficient number of extracted, sound human teeth is increasingly more difficult. Therefore, it is wise to plan for the most efficient use of available teeth. Blindly using a particular sample size without statistical justification is to be avoided, particularly when rarer, flat, uncut enamel and cementum substratesare needed. A pilot study using five specimens usually provides enough data to determine the variability within the data to establish a statistically valid sample size. Besides performing this calculation, a good statistician should be consulted prior to conducting the pilot study because (s)he may be able to suggest ways of increasing the power of measurement to reduce the sample size before the pilot study is even started. Depending on the researcher's or statistician's approach to research design,there is often the temptation to use larger sample sizes than necessary to compensate for lost or defective specimens. This is generally not a good idea unless the researcher knows in advance that a particular specimen preparation technique is very sensitive to uncontrollable variables.

Step 2: Tooth Selection and Manipulation

For studies involving uncut enamel substrates (veneers, mini-restorations for incisal edges, shade blending for purely esthetic reasons, and recontouring) and uncut cementum substrates (root-surface restorations and tooth desensitizing procedures), the crowns of upper and lower central and lateral incisors should be reserved. The "flat" surfaces of roots from all teeth should also be reserved. The bonding area used with the method being presented in this series of articles is 2.3798 mm in diameter. This is a fairly small area that allows relatively "flat" areas of curved surfaces to be utilized. Generally speaking however, the variability within data will be greater for these studies because the imperfect surfaces introduce physical, chemical, and mechanical variations that can ultimately affect bond strengths. Therefore as a general rule, larger sample sizes may be necessary when studying bonding to uncut enamel and cementum substrates. Once the uncut substrates have been used, they may be recycled as cut substrates. This is easily accomplished by grinding away the surface of the crown or root to create either cut enamel and/or cut dentin.

When selecting extracted teeth for a particular study, separate sound teeth from unsound and previously restored teeth. Do not discard unsound and previously restored teeth without assessing all possible bonding sites. Virtually all extracted human teeth (sound, diseased, traumatized, or previously restored) may be

used to study various cut substrates with the test method being presented. For example, because of the relatively small bonding area required, different areas of a tooth crown can be used to study the effects of differently oriented enamel rods or different densities of dentin tubules (Figure 1).

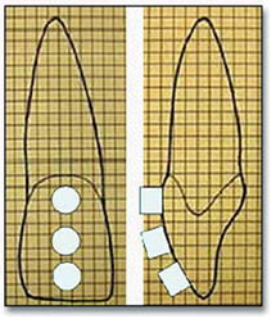

Figure 1. Possible bonding sites on a maxillary central incisor. Note how different orientations may be achieved.

The variations possible for bonding studies are limited only by how the tooth substrate is positioned within the embedding mold. It must be kept in mind that bonding to an unsound tooth structure, whether accidental or planned, is not an uncommon event clinically but has very little scientific justification based on bond strength studies.[5] Bonding to tooth surfaces previously in contact with metallic restorations or dental cements has not been studied even though such information is extremely important in a modern clinical practice (Figure 2).

The effects of materials from previously restored teeth on subsequent bonding can be studied after the failed restoration has been removed. The effects on bonding with substrates that have

Figure 2. Split tooth with large amalgam restoration has suitable has suitable bonding site (upper left). Root portion of tooth has been removed by grinding (insert). Specimen is aligned in embedding mold with sticky wax so that bonding surface is nearly parallel to mold surface (upper right).

sclerotic dentin or reparative dentin may be studied (Figure 3). The effects of fluoride treatments, cleansing and disinfecting procedures, and desensitizing treatments can be studied. The effects of etchants, etching times, rinsing times, degrees of drying after rinsing, as well as the effects of cements and liners on bond strengths can be also be examined.

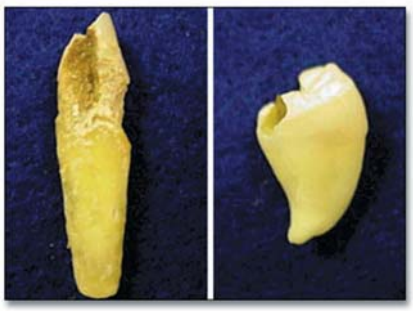

Figure 3. Tooth substrates with unsound tissue, scierotic tissue, or reparative tissue may be tested with this method.

Step 3: Tooth Preparation

When preparing the extracted teeth, remove any residual soft or hard tissues (Figure 4).

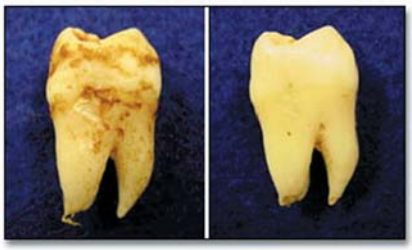

Figure 4. All residual soft and/or hard tissue must be removed from the specimens. Special care should be taken when cleaning unprepared cementum surfaces to prevent damage.

Take great care when doing this if an underlying cementum surface will be used. Carefully remove any metallic restorations without damaging the adjacent enamel or dentin structures (Figure 2). Remember a relatively “flat” surface, at least 2.3798 mm in diameter, is required. Once each tooth has been assessed, unwanted crowns or roots may be removed using a grinding wheel on a cast trimmer (Figure 5).

When both the crown and the root need to be saved, a diamond saw should be used. Multiple roots may be separated, but generally no other preparation is required. Crowns may be sectioned in half. This may be done by placing the

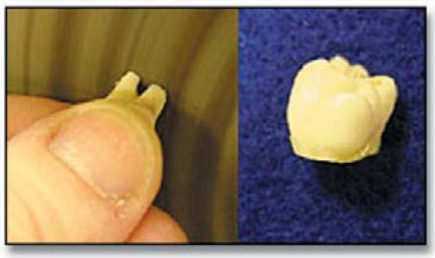

Figure 5. A coarse grinding wheel may be used to remove unwanted roots or coronal parts of a tooth. A diamond saw may be used when several different parts of the tooth are wanted.

widest part of the crown in a diamond saw so the long axis of the tooth is parallel to the blade. This can be quite difficult because if the crown should slip during sectioning, it is likely the saw blade may be damaged. Very small, parallel, flat surfaces may be ground on opposite sides of the crown to help stabilize it in the saw.

Step 4: Embedding Procedure

The embedding mold used for this method is made of polycarbonate and does not stick to methylmethacrylate resins. Therefore, no separating medium is required. The mold (Ultradent Products Inc., USA) can make 15 cylindrical specimens, each 1 inch in diameter and up to 1 inch in height (Figure 6).

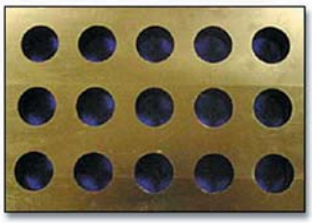

Figure 6. A polycarbonate embedding mold, supplied by Ultradent Products Inc., makes 15 specimens without the need for a separating medium.

The mold is prepared by applying 1½ inches of clear cellophane tape to close one end of the specimen holes (Figure 7).

Figure 7. Transparent cellophane tape closes one end of the specimen mold and makes it possible to see the specimen to adjust the orientation of the specimen in the mold hole.

Care should be taken to remove any air pockets around the edges of the specimen holes to prevent leakage of the embedding resin. Each tooth substrate is positioned within its specimen hole using a small portion of sticky wax, if necessary to secure uncut specimens, as well as the exposed adhesive on the cellophane tape (Figure 8).

Figure 8. The correct orientation of the specimen is virtually assured because the bonding surface can be clearly seen through the clear cellophane tape. A small amount of sticky wax may be used to refine the specimen orientation.

The precise orientation of each substrate can be observed through the clear cellophane tape. This enables specific substrate orientations to be achieved for various test procedures. Because of the design of the polyethylene insert used to "restore" the tooth substrate, a 1 inch diameter rubber washer should be placed into the specimen hole when uncut specimens are to be embedded (Figure 9). Once the washer is removed after embedding, the additional space makes it easier to get close adaptation of the polyethylene insert to the more irregular uncut surfaces.

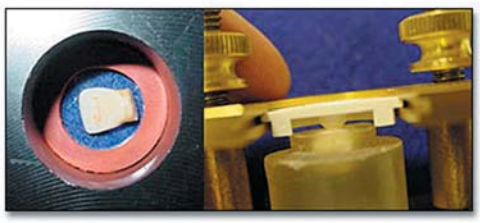

Figure 9. A 1" rubber washer may be inserted into the specimen mold to provide space for the polyethylene insert when restoring more irregular unprepared substrates.

Clear embedding resin is mixed with a fluid consistency. Clear, pink Orthoresin (Dentsply DeTrey, Germany) mixed in 1 part powder/4 parts liquid provides ample pouring time and allows for specimen observation. Some resins do not shrink as much as others and may make it difficult to remove specimens from the mold. Some resins bloat and overflow when mixed at a 1:4 ratio. Some opaque resins work well but prevent specimen observation. These resins are to be avoided. Once specimens have hardened, they may be removed with simple finger pressure.

Step 5: Final Specimen Preparation

For uncut substrates, if sticky wax was used to position the substrate, the sticky wax must be completely removed at this time. The acrylic embedding resin on both ends of the specimen

must be made parallel by rotary grinding with coarse and fine wheels on a cast trimmer modified with a grinding apparatus (Figure 10). Great care must be taken not to alter unprepared enamel or cementum substrates. Generally, no other preparation is required unless the research protocol calls for additional treatment, such as fluoride, cleansing, disinfecting treatments.

Figure 10. Both ends of the specimen must be ground parallel to each other using coarse and fine grinding wheels. A special rotating grinding apparatus is mounted on a cast trimmer. It should be noted, with unprepared substrates, only the opposite end of the specimen is ground.

For cut specimens, the specimen is positioned in the grinding apparatus. The enamel or cementum surface is removed creating either a cut enamel or cut dentin surface. Again, no other preparation is required unless the research protocol calls for additional treatment.

Tooth Restoration and Debonding

At this point, the substrate is ready for the next steps of the test method: the restoration and debonding procedure.

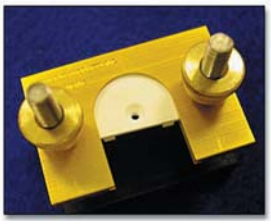

Figure 11. Bonding clamp with polyethylene bonding mold insert.

Step 1a: Direct "Restoration" of Tooth Substrate

A bonding clamp with bonding mold insert is used to "restore" the tooth substrate (Figure 11). Prior to placing the tooth substrate specimen into the bonding clamp, any pretreatments prescribed for the research protocol should be applied. Generally, such pretreatments include washing, conditioning, and applying bonding agents. A basic bond strength test protocol would include rinsing with oil-free water from an air/water syringe for 5 seconds, applying and agitating etchant for 15 seconds, rinsing with an oil-free air/water syringe for 5 seconds, blot drying with lint-free gauze to remove puddles of water, and applying a bonding agent.

When the specimen is placed in the bonding clamp, great care should be taken not to over-tighten the clamps. Very light friction should be applied to the tightening screws (Figure 12). The bonding mold insert has a very delicate knife edge that can easily be deformed if over-tightening occurs. This is especially true for more irregular uncut substrates. Each insert may be used repeatedly. At some point when the insert becomes more difficult to slide off the "restoration," the insert is replaced. Generally, this may be after 20-25 uses, depending on how carefully the researcher has handled the insert.

Figure 12. Great care must be taken when positioning and securing the polyethylene bonding mold insert so as not to damage the knife edge.

Once the specimen has been placed in the bonding clamp, the "restoration" may be applied (Figure 13). Depending on the research protocol, one composite should be used when testing bonding agents. Generally, shade A2 is dispensed from unidose cartridges to reduce statistical variability. The "restoration" mold is 2.3798 mm in diameter and 2 mm in height. The insert is designed this way to provide a constant "restoration" size and a constant curing light position.

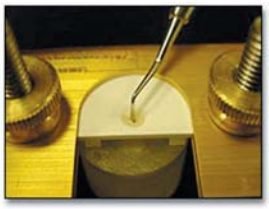

Figure 13. Generally, "unidose" capsules should be used to begin filling the insert mold.

Conveniently, a 1 lb load equals 1 MPa with this specimen size. A 1 mm condenser should be used to place the composite below the bevel on the mold (Figure 14).

Figure 14. A 1 mm condenser tip is used to press the restorative material into the insert mold below the bevel. Failure to place the material below the bevel will make it impossible to remove the specimen from the mold

Care should be taken to assure the condenser does not scrape the sides of the mold and that no composite extends onto the bevel. The latter would make removal of the specimen impossible. Once light-initiation of the "restoration" is completed, the screws on the bonding clamp should be loosened while holding the specimen mold and specimen in place (Figure 15). A condenser tip is positioned on the restoration and light pressure is applied to the specimen while the bonding clamp is lifted clear of the specimen (Figure 16). A sharp #12 scalpel is used to gently remove flash without disturbing the "restoration."

Figure 15. Light initiation is completed.

Figure 16. The clamp screws are carefully loosened. A condenser tip is used to gently hold the specimen in position as the insert is lifted up.

Step 1b: Indirect "Restoration" of Tooth Substrate

There are many instances where direct restorations are not indicated for tooth restoration. In these cases, immediate bond strengths for resin-bonded indirect restorations are as important as for direct restorations. Indirect "restorations" may be easily made by positioning the polyethylene mold insert on a glass slide, placing the restorative material in the mold, curing the restoration, and then cementing the indirect "restoration" on the tooth substrate using a pressure balance. The balance is used to apply a constant pressure to the "restoration" during cementation. Specially-processed "restorations" may also be cemented using the same technique, allowing for possible differences in specimen size.

Step 2: Debonding the "Restoration"

Immediately after the specimen has been removed from the bonding clamp, it is placed in the test base clamp and positioned in an Instron or other mechanical testing system. This may be done by lowering the crosshead contact to a position slightly below where the "restoration" is able to slide into the contact notch (Figure 17).

Manually raise the crosshead until the "restoration" is just able to slide into the notch. Position the specimen so the substrate is flush against the crosshead (Figure 18).

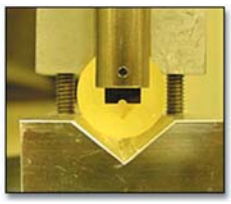

Figure 17. The crosshead knife is lowered to just below where the specimen slides into the notch.

Figure 18. The substrate is flush against the crosshead knife.

Again, uncut substrates may not be perfectly flush and may cause variations in data. The crosshead rate of testing should be 1 mm/min but slower and faster rates may be used. Peak load is usually recorded for this test method, but elasticity, resilience, and toughness may also be calculated. As noted before, the "restoration" diameter automatically allows a conversion from 1 lb to 1 MPa.

Once debonding has occurred, the specimen is removed from the testing machine. Recycling is encouraged within the limits of the test specimen by grinding the substrate surface away and preparing for another test. Of course, surface failure modes should be documented according to the research protocol.

Conclusion

This article completes the step-by-step method that can be adapted to many different bonding test protocols. It is hoped a standardized testing protocol will make it possible for more uniform data to be reported that is more valuable to the practicing clinician.

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