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Adhesion to tooth structure: A critical review of "micro" bond strength test methods

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ABSTRACT

The objective of this paper is to critically review the literature regarding the mechanics, geometry, load application and other testing parameters of "micro" shear and tensile adhesion tests, and to outline their advantages and limitations. The testing of multiple specimens from a single tooth conserves teeth and allows research designs not possible using conventional 'macro' methods. Specimen fabrication, gripping and load application methods, in addition to material properties of the various components comprising the resin–tooth adhesive bond, will influence the stress distribution and consequently, the nominal bond strength and failure mode. These issues must be understood; as should the limitations inherent to strength-based testing of a complicated adhesive bond joining dissimilar substrates, for proper test selection, conduct and interpretation. Finite element analysis and comprehensive reporting of test conduct and results will further our efforts towards a standardization of test procedures. For the foreseeable future, both "micro" and "macro" bond strength tests will, as well as various morphological and spectroscopic investigative techniques, continue to be important tools for improving resin–tooth adhesion to increase the service life of dental resin-based composite restorations.

1. Introduction to the review

Bond strength testing has been traditionally accomplished by creating one test specimen per tooth or tooth surface which is then loaded to failure in either shear (SBS), tensile (TBS), or fracture-based manner. Currently, the most common approach is to load multiple test specimens from each tooth in either a tensile (μTBS) or shear (μSBS) manner. The earliest reference to the use of a 'micro' tensile device for biomechanical engineering was by Okuno who studied whisker-reinforced metal alloy [1]. Subsequently microtensile testing was used to evaluate tensile strength of collagen and elastin fibers in porcine tricuspid leaflets [2], aortic valves [3], patellar groove articular cartilage [4], and cortical bone [5].

Sano et al. introduced microtensile testing to dentistry to measure the ultimate tensile strength and modulus of elasticity of mineralized and demineralized dentin [6]. This test

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offered versatility that could not be achieved using conventional shear and tensile methods. This groundbreaking work was then applied to bond strength evaluations, such as; normal *vs.* caries-affected occlusal dentin [7], normal *vs.* sclerotic cervical dentin [8], different regions of restored MOD preparations [9], *in vivo* class V restorations [10], regions of occlusal dentin [11], dentin depth and cavity configuration [12], and hundreds of other dental biomaterials studies. "Micro" tensile bond strength (μTBS) lends itself to additional research designs that the "macro" tests do not, such as, the elimination of tooth dependency through balanced designs [13], and has shown reduced test variance [14]. Simply using a smaller mechanical bond strength test specimen in the laboratory does not eliminate the challenges that teeth, beyond their relatively small size, present, i.e., structural heterogeneity, mechanical anisotropy, visco-elasticity, presence of dentinal fluids, and simulation of their complex and harsh functional environment.

A 1995 review [15] of dentin adhesion testing stated a number of advantages for the μTBS approach: more adhesive failures and fewer cohesive failures, measurement of higher interfacial bond strengths, means and variances can be calculated for single teeth; permits testing of irregular surfaces; permits testing of very small areas and facilitates SEM/TEM examinations of the failed bonds since the surface area is approximately 1 mm^2. Drawbacks cited were the labor intensity, technical demand, and dehydration potential of these smaller samples. In an excellent follow up paper by Pashley et al. the versatility of microtensile methods over conventional approaches to evaluate clinically relevant sites and substrates was comprehensively detailed and critiqued [16].

Microtensile bond strengths tend to be much higher (often 2× to 4×) than that of macro TBS values because the defect concentration in the small cross-sectional interfacial areas is lower [14,17]. More recently, the μSBS test has become popularized as an alternative to the conventional SBS test [18,19]. The μSBS test again obtains multiple specimens per tooth but without the damage history due to specimen sectioning, trimming and shaping that is required with μTBS testing. MicroSBS, as with μTBS, has been advocated as a method for regional mapping, but also is very conducive to depth profiling of different substrates.

Currently no broad agreement exists within the scientific community as to the appropriate conduct, usage and interpretation of these tests and any attempts at standardization of test methods have been difficult. The objective of this paper is to critically review the literature regarding the mechanics, geometry, load application, preparation effects, and other testing parameters of these "micro" adhesion tests, outlining their advantages and limitations.

2. Literature

2.1. *What makes a good bond strength test?*

The realization of a widely accepted, validated, standard test method for adhesive resin–tooth bond strength testing is an elusive and controversial endeavor [20–22]. Although a consensus or standard approach does not currently exist in dentistry, bond strength testing remains useful and necessary for the screening of new products and study of experimental variables. Therefore, the scientific community should strive to identify important testing parameters and journal editors should demand comprehensive reporting of these parameters so that critical interpretations of data and meaningful comparisons of results can be achieved. Published studies, test standards (see Appendix A), and technical reports [23] can be referenced for important considerations regarding specimen fabrication, test conducting, and reporting. First; however, basic principles regarding the use of strength-based testing must be understood.

Strength-based testing does not quantify an inherent material property of the bond of restorative materials to tooth structure. The measured bond strength and the failure mode or debond pathway produced is dependent, among other things, upon: flaws existing within or between materials, specimen size and geometry, material properties of each component of the bonded assembly, and method of load application. Adhesive resin–tooth bond strength testing involves two separate substrates and complicated interphases or zones of interdiffusion between these components, all possessing different material properties. Therefore, even if a perfectly uniform tensile load could be applied across a resin-based-composite (RBC)–dentin adhesively bonded joint a non-uniform stress distribution will occur in the adhesive joint. The ratio of Poisson's ratio (ν) to the Young's modulus (E) would have to be equivalent for the: RBC, RBC–adhesive resin 'interface', adhesive resin, adhesive resin–hybrid layer 'interface', hybrid layer, hybrid layer–dentin 'interface', and dentin to produce a uniform stress state [24]. In other words, the RBC and dentin substrates and the adhesive resin and their interdiffusion zones under loading, would all have to either: (1) not deform laterally, or (2) deform laterally in an equal manner, to permit a homogenous stress distribution. The ratio ν:E is higher for the adhesive and hybrid layer than the adherends, thus the lateral deformation in these regions will be greater than for the RBC and tooth structure, if the bond remains intact under loading, a complex stress distribution will occur in the adhesive resin and hybrid layer. Clearly, a complex stress state exists in 'macro' and 'micro' shear and tensile testing of the adhesive resin–tooth bonded joint.[2] These prior comments ignore the material property differences that commonly exist within a single material when comparing bulk *vs.* surface regions. As pointed out at an earlier ADM meeting [25], it may be more appropriate to refer to these tests, regardless of type of test or size of specimen, as simply "bond strength tests".

Smaller test specimens are 'stronger' than larger ones due to the lower probability of having a critical sized defect present and aligned in a crack opening orientation relative to the applied load. Tooth structure and dental adhesives are relatively brittle materials, therefore, the Griffith strength

[2] Also why both K_{Ic} and K_{IIc} loading modes must be considered in the determination of interfacial fracture toughness or alternatively, to use a fracture energy (G_c) approach, due to mode mixity across RBC–tooth adhesive joints.

Table 1 – Dentin SBS results using Ultradent jig loaded at 1 mm/min. Mold diameter is 2.38 mm for a cross-sectional bonding area of 4.45 mm^2. Groups identified by different letters are significantly different at $p < 0.05$ (unpublished data).

Specimen fabrication method	SBS (MPa ± SD)	Predominate failure mode
Adhesive system applied and light-cured *before* mold placement	34.08 ± 2.45 A	Cohesive in dentin 67%
Adhesive system applied *before* and light-cured *after* mold placement	32.06 ± 1.36 B	Cohesive in dentin 83%
Adhesive system applied and light-cured *after* mold placement	22.92 ± 1.28 C	Apparently adhesive 83%

equation [26]:

$$\sigma_f = \left(\frac{2E\gamma}{\pi c_o}\right)^{1/2}$$

where σ_f is the fracture strength in uniform tension, E the plain strain elastic modulus, γ the surface energy per unit area, and c_o the starter crack size, applies. This reminds us that the measured "bond strength" at failure will be dependent not only upon the fracture strength but the presence of flaws [27]. Another way to think about this is that the resultant bond strength data obtained from mechanical testing represents a statistical distribution of the flaws (discontinuity, defect, bubble, void, residual solvent, etc.) present in each test specimen leading to its failure. This concept lends itself nicely, not only, to the presentation and interpretation of data by probabilistic statistical methods such as Weibull, but as an explanation as to why smaller specimens are stronger. Another way to say this is "Strength values (whether from testing a monolithic specimen or a bonded specimen) simply provide insight into the stresses a particular material will support given the flaw size distribution" [27]. Griffith demonstrated that drawn glass fibers were "stronger" than ordinary glass, proving the volume dependency of strength, as did da Vinci, four centuries earlier, when testing wires of various lengths. This volume dependency of strength tells us that a smaller test specimen will be less likely to have a larger flaw that leads to its failure. Therefore a higher apparent "strength" is measured. In converse, the larger the size of the specimen, the more likely will be the presence of a larger flaw that leads to its failure, and, therefore, a lower apparent strength. The adoption of the term "microtensile" into our literature does not fully acknowledge these principles. Tensile testing should not be simply categorized as those with a less than 1 mm^2 (microtensile) or greater than 1 mm^2 cross-sectional testing region without full consideration of this volume dependency of strength. Nevertheless, use of the term "microtensile" is firmly established in our literature.

3. Microshear bond strength testing

Shear bond strength testing with bonded cross-sectional areas of 1 mm^2 or less is also referred to as 'micro' SBS [18]. This relatively simple test permits efficient screening of adhesive systems, regional and depth profiling of a variety of substrates, and conservation of teeth. Aqueous storage durability studies are also possible with μSBS due to the relatively short diffusional distances (0.02–0.05 mm) from the cavosurface [28]. The general findings based upon FEA and failure mode analysis of macroSBS testing; however, hold true for μSBS testing. These FEA findings being: (1) tensile stresses produced by the bending moment at load application being responsible for fracture initiation, (2) highly nonuniform stress distribution concentrated in the substrate, and (3) a nominally measured bond strength that severely underestimates the true stress the specimen resisted at fracture [29–32].

In addition, Placido et al. concluded that μSBS results may actually worse represent shear bond strength than the conventional macroSBS test [19]. Increased stress concentration and tensile forces during shear load application were shown when factoring in the relatively thicker adhesive layer, farther load application from the adhesive bond, and the use of lower modulus flowable RBCs (to avoid the introduction of flaws in the small molds required) common to μSBS tests. In contrast, McDonough et al., using three-dimensional FEA demonstrated that the tensile forces during loading could be minimized by optimizing specimen dimensions and load application location [33].

A significant advantage over μTBS methods is that the μSBS specimen is pre-stressed prior to testing only by mold removal. Similar to macroSBS methods; however, the use of the mold for RBC placement can lead to the introduction of flaws and different stress concentrations upon shear loading [34]. Adhesive resin flash beyond the restoration margin will be present if the adhesive is cured before mold placement and if the adhesive bond is formed within the mold a greater chance of uneven adhesive resin thickness, meniscus formation at the border and introduction of flaws are possible. These specimen fabrication methods should be thoroughly described as they can significantly affect results (Table 1). Regardless, μSBS methods remain an especially useful test for those substrates with properties such as glass ionomers or enamel, that make them particularly susceptible to the specimen preparation effects and testing conditions of μTBS testing.

4. Microtensile bond strength testing

The microtensile bond strength test is calculated as the tensile load at failure divided by the cross-sectional area of the bonded interface. However this nominal strength is valid only if a state of uniform, uniaxial stress is present [35–37] with the maximum tensile stress present and homogenously distributed in the region of the smallest cross-sectional bonded area [38]. Therefore, if experimental conditions are constant, it is expected that μTBS results for restorative material A bonded to substrate B from different laboratories would be in agreement. However, variable methods and parameters have been employed by the different laboratories all over the world resulting in bond strength data that can hardly be compared across studies [38,39].

Several factors potentially contribute to this interlaboratory variability and are nearly impossible to identify from published papers. Searching PUBMED (January–July 2009) with the keyword "microtensile" resulted in more than 90 papers.

Only 10% of these papers described in any detail: the gripping device, specimen fixation method to this gripping device, mechanical properties of the adhesive and the adherends tested, or specimen shape. Different results could be obtained with any change in these testing variables, making interlaboratory comparisons of little value. Without full reporting of methods, a simple rank listing of groups by load at breakage would be just as useful for comparisons.

Advantages of μTBS testing include [15,16]:

(1) Conservation of teeth,
(2) Evaluation of regional bond strengths possible [11,40,41],
(3) Evaluation of remaining dentin thickness effects possible [42,43],
(4) Evaluation of intra- and inter-tooth variability [9,44],
(5) Evaluation of bond strength to various cavity walls in restoration possible [45,46],
(6) Evaluation of bond strength to intra-radicular dentin is possible,
(7) Conducive to evaluation of the effects of RBC polymerization shrinkage stress [12,47],
(8) More uniform loading may be possible due to less bending offset, relative to conventional tensile testing, due to alternative gripping method,
(9) Fewer cohesive failures in substrates,
(10) Bond strengths are higher than those measures from conventional tensile and shear bond strength tests due to the decreased number of defects in the substrate or at the bond interface (advantage?),
(11) Additional research designs can be performed to account for tooth dependency, such as, multiple surfaces within a cavity, various substrates within a tooth, durability testing by aqueous storage [13],
(12) Accelerated environmental aging is feasible by aqueous storage due to short diffusional distances [48],
(13) Possible to evaluate very small surface areas when necessary, e.g., caries-affected dentin [7],
(14) Can minimize shear effect by tensile testing a relatively flatter region of tooth when not preparing surface, e.g., unground enamel [49,50],
(15) SEM fractography can be readily performed to determine the mode of failure [51],
(16) Clinically retrieved restorations can be evaluated [10,52,53],
(17) Conducive to comprehensive examination of research question, such as, mechanical, morphologic and chemical studies on same sample [54,55].

Limitations of μTBS testing include [15,16]:

(1) Labor intensive, technically demanding,
(2) Difficult to measure very low bond strength (<5 MPa),
(3) Specimens easily dehydrate,
(4) Specimens easily damaged,
(5) Post-fracture specimens can be lost or damaged when removing from active gripping devices that use glue,
(6) Difficult to fabricate with consistent geometry, surface finish and damage history without aid of special equipment,
(7) Lack of consensus exists for conduct of test, reporting of pre-test failures and fractures outside of the designated testing region of specimen (applicable to all dental bond strength testing).

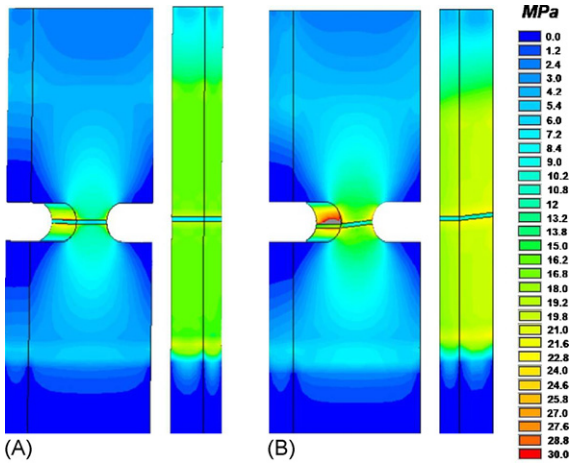

Fig. 1 – von Mises stress distribution. (A) Hourglass- and stick-shaped specimens with bond line perpendicular to load; (B) hourglass- and stick-shaped specimens with angled bond line.

4.1. *Gripping devices*

Surface preparation of dentin and enamel for μTBS testing should produce a flat surface with a standardized, clinically relevant smear layer. This specimen should then permit the tensile load to be applied perpendicularly to the adhesive bond line. Regardless of the method to produce this surface preparation, such as, dental handpiece or SiC polishing paper, it is not uncommon to produce either a non-perpendicular or non-flat surface or both. This can produce spurious μTBS data due to: inaccurate bonded cross-sectional areas, incorporation of additional flaws, and a non-normal load application. A non-normal load application, whether due to the specimen or gripping mechanism significantly alters the stress distribution at the bonded interface [37,39]. Several specimen gripping devices, both active and passive, have been developed in an attempt to properly apply a tensile load normal to the bond line [13,56,57], this will occur; however, only if the specimen's bond line is properly aligned with its gripping surfaces (Fig. 1).

Test specimens are attached to the load train couplers of mechanical testing machines by either active or passive gripping devices. Active gripping can be either mechanical or, most commonly in μTBS testing, via fast-setting glue. These specimen-fixation procedures require careful manipulation and special test jigs [6,8,41,56–58]. Theoretically, these jigs should assure that a pure tensile force is applied to the test specimen. Bending forces can occur during load application due to: non-parallel specimen alignment, bond line not perpendicular to the specimen gripping surfaces, and/or, uneven gripping forces [31,37,59].

In the original microtensile test set-up the specimens were glued onto a Bencor Multi-T gripping device with quick setting cyanoacrylate gel that covered the entire surface of both specimen ends [6]. The Ciucchi's jig was later introduced as

Table 2 – Mode of failure and fracture location using three different gripping methods for dentin μTBS testing. Dumbbells with 0.5 mm² cylindrical cross-sectional area, 1 mm gauge length, and 0.6 mm radius of curvature were used for the Dircks device. Sticks with 1 mm² cross-sectional areas were used for the Geraldeli's jig (unpublished data).

Gripping device (# tested)	Interfacial	Cohesive in dentin		Cohesive in resin-based composite		Mixed	
		Gauge region or test area	Non-gauge region or involving glue	Gauge region or test area	Non-gauge region or involving glue	Gauge region or test area	Non-gauge region or involving glue
Dircks device [40]	26	9	2	0	0	3	0
Geraldeli's jig with Loctite [37][a]	14	1	10	1	6	5	0
Geraldeli's jig with Zapit [36][a]	7	0	9	5	3	12	0

[a] Seven total specimens were excluded due to either glue on interface or broken while handling.

a modification to this active gripping method [8]. Neither of these devices guarantees proper alignment because the specimen is glued to a flat gripping surface [57,59]. A groove parallel to the applied load was added in the so-called, Geraldeli's jig, in an attempt to improve specimen alignment [56]. Later, the Ciucchi's jig was modified in a similar manner for the same reasons [57]. Relative to a flat faced active gripping device, a notched gripping device: requires less glue, reduces the possibility of glue contamination on the bond line, and reduces test variability for both stick and dumbbell specimens [42,57].

Regardless, the use of glue for active gripping of micro-specimens is technically challenging and specimens must commonly be reglued during testing due to either specimen pull-out or glue detachment from the face of the gripping device. Additionally the particular glue used or unknown forces imparted upon the test specimen during curing of the glue may affect results (Table 2). Our laboratory previously used Jacobs' chucks aligned in the upper and lower members of a displacement controlled testing machine to grip μTBS specimens. The specimen was first fixed to an acrylic rod with RBC, trimmed, placed in the Jacobs chuck and lowered into an empty Plexiglas rod below for fixation with cyanoacrylate. A fixed period of time was allowed for the cyanoacrylate to harden before tensile testing; during this time, compressive forces then rapidly increasing tensile forces were recorded on the load cell. Not uncommonly the specimen had to be reglued multiple times before testing. These experiences, combined with the difficulty of forming the specimen notch manually, lead us to eventually develop a self-aligning, glueless, passive gripping device, i.e., "Dircks device" (Fig. 2). This methodology requires computer numerically controlled (CNC) machining to produce a dumbbell-shaped specimen with geometry that permits uniform contact between the gripped section of the specimen (neck region) and the grip faces of the Dircks device [60]. This passive gripping device consists of two identical aluminum parts guided by two vertical stainless steel alignment pins. A central notch or pocket, with a radius of curvature (0.6 mm) in the lateral shoulders matches that of the specimen. The main advantages of this device are: efficient and reproducible specimen alignment, simple manipulation without the use of any glue, lack of specimen dehydration, remaining dentin thicknesses as small as 1 mm can be tested, and absence of preloading stresses possible with active gripping methods. Moreover, the dumbbell-shaped micro-specimens used for this device have been shown to present a better stress distribution than hourglass or stick-shaped specimens [61,62].

Poitevin et al., developed a μTBS testing device with top-bottom fixation to minimize stress concentrations [42,57]. A trimmed dumbbell-shaped specimen is mounted by one end in a pin-chuck and lowered onto a horizontal table for fixation of the opposite end with cyanoacrylate. This top-bottom set-up demonstrated slightly less variability, a higher number of apparently interfacial failures, and a more homogeneous fracture surface, when compared to specimens tested using a flat jig. These results are attributed to a more perpendicular alignment of the interface and a stress impact more completely following the specimen's long axis, i.e., less bending offset. They also demonstrated that the use of a notched jig diminishes the intra- and inter-tooth variability, as compared

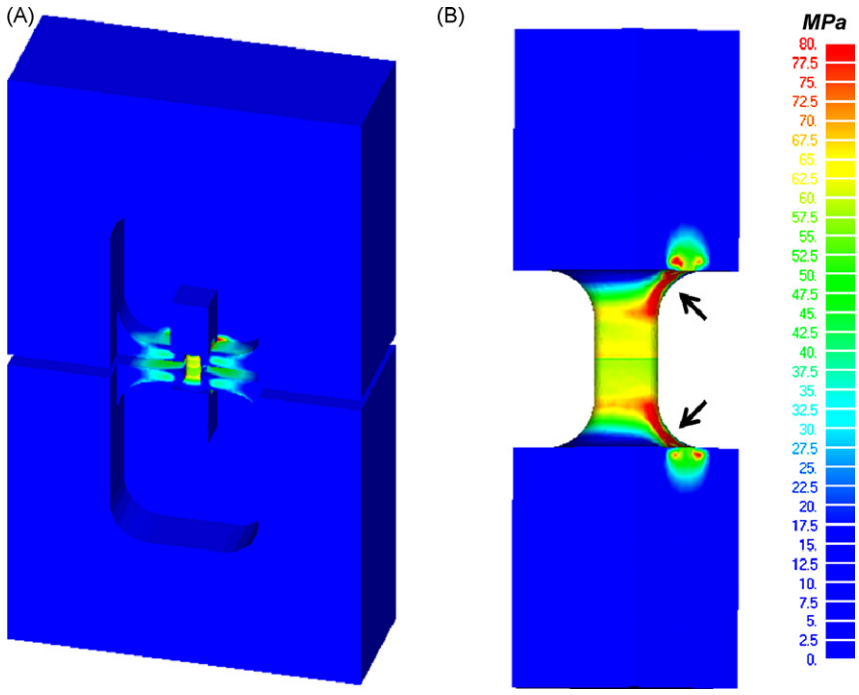

Fig. 2 – von Mises stress distribution (A) μTBS testing set-up of Dircks device; (B) dumbbell-shaped specimen tested in the Dircks device showing homogeneous stress distribution in the adhesive bond region; arrows indicate contact area between Dircks and the dumbbell specimen (unpublished data).

to a flat jig. Regardless of active or passive gripping or the particular gripping device, standardization of the intra- and inter-tooth differences can be partially addressed by using a fixed number of specimens per tooth, minimizing regional differences, and using a fixed number of teeth per group [63]. Soares et al., verified with FEA analysis that increasing the number of faces of specimen fixation was directly related to stress homogeneity, with the best distribution occurring when all surfaces were attached [38] (Fig. 3).

The use of glue as a means of active gripping has the advantage of being able to attach relatively fragile and non-geometric test specimens, but also has several potential limitations that may affect results [38]:

(1) Non-static loads to the specimen during glue polymerization,
(2) Effects on specimen geometry dependent load distribution [62],
(3) Some degree of specimen drying is required for attachment,
(4) Glue components may come into contact with the specimen interface or testing region during application (Table 2),
(5) Glue may possess inadequate attachment strength, leading to repeat testing or an inability to test,
(6) Fractures may occur near or in glue attachment, and not in the testing region (Fig. 4),
(7) Glues deform before rupture due to relatively low Young's modulus [64],
(8) Time consuming,
(9) Difficult to remove fractured specimens from device without damage to or loss of the specimen,
(10) Difficult to remove remaining glue from device in preparation for next test,
(11) Minimum substrate size required for glue application [58].

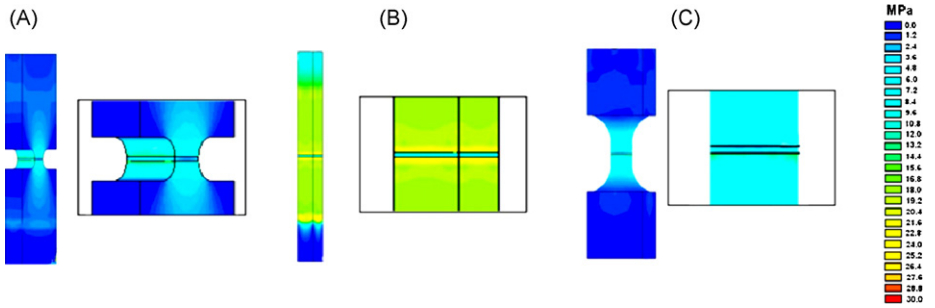

Fig. 3 – von Mises stress distribution in uTBS specimen geometries when gripped on all surfaces (A) hourglass; (B) stick; (C) dumbbell [38].

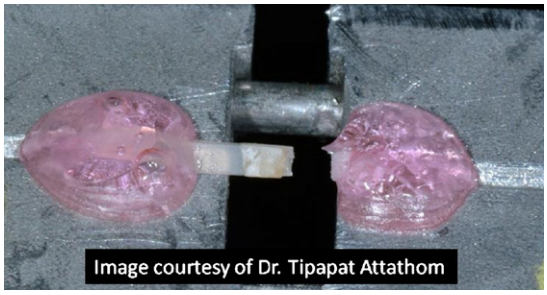

Fig. 4 – Post-tensile test resin-caries-affected-dentin specimen in Geraldeli's jig using Zapit cyanoacrylate showing cohesive fracture in the dentin located at the interface of specimen and glue.

Due to limitations with the use of glue, our laboratory uses, as described earlier, a non-gluing, passive gripping device. This; however, requires additional equipment, that is cost-prohibitive for wide adoption, to shape the specimen for uniform contact between the gripped section of the specimen and the grip faces [18,47,60,65]. Active gripping methods, such as glue or clamps, are then required if tensile specimens cannot be precisely machined. If dumbbells are not feasible, the use of stick-shaped specimens due to the high stress concentration factors found with notched hourglass geometries is encouraged [38,39].

4.2. *Specimen geometry and preparation effects*

The first specimen design for μTBS proposed by Sano et al. was the hourglass shape. A sharp notch of the hourglass was designed to concentrate stresses where the adhesive bond interface is located, avoiding the undesirable fracture of the adherends near the glue [6]. Several specimen types have been subsequently reported that can be categorized as; hourglass, slab (rectangular), stick (square), and dumbbell geometries, with cross-sectional shapes that are either square, rectangular or round. The specimen geometry has a significant influence on homogeneity of stress and if stress concentrations cannot be completely avoided, they should be at least minimized. The stress concentration and pattern in the hourglass samples is very different from the dumbbell and stick samples [38]. The hourglass sample fails at lower stress compared to the stick or dumbbell, due to the large stress concentration induced in the adhesive [36,39]. The stress concentrates at or very close to the adhesive edges of the hourglass specimen indicating where the initiation of failure is likely; different types of failures for the hourglass specimen, compared to dumbbell and stick, have been reported [38,39].

A basic tenet of materials testing is to produce consistent test specimens. For the hourglass-shaped specimen design, there are a large number of designs in the published literature [16], therefore, caution is advised in interpreting and comparing results [38,66]. The hourglass neck geometry should be carefully analyzed since the radius of curvature of the notch has a significant influence on stress concentration values [67]. The use of a equipment specially designed for notch formation results in more uniform geometries of consistent size with potentially less stress imparted to the specimen than manually trimming [18,65]. This equipment is most often used to prepare dumbbell-shaped specimens [48,65,68]. Dumbbell-shaped specimens demonstrate peak stress concentration in the radius of curvature or 'neck' (Fig. 2) which could lead to failure in the non-gauge region during testing, especially problematic for resin-enamel testing (Table 3) [62].

Through all phases of specimen fabrication, a careful examination should be carried out to identify any defects that, based on pre-determined criteria, would exclude the specimen from testing. Appropriate magnification should be used to determine failure location (gauge or non-gauge) and debond pathway (cohesive in substrates, apparently interfacial, or mixed). Any failures located outside of the gauge region should be treated as right-censored data points. These right-censored data points potentially had higher bond strengths than the recorded nominal strength and bias will occur if excluded. We have used dumbbell-shaped resin–dentin specimens in our laboratory for several years and have very rarely observed a fracture location entirely outside of the test gauge region. Those failures that occur in the specimen neck (radius) may be due to stress concentration (Fig. 2), gross voids between RBC increments or inadequate remaining dentin thickness. Significantly different fracture locations and failure modes are commonly observed with the use of active gripping with glue (Table 2).

However, a higher incidence of non-gauge failure location occurs, as does pre-testing failures (PTF), when testing resin-enamel bonds with the passively gripped dumbbell specimen (Table 3). This may be due to the material property differences in the enamel substrate as compared to dentin. When subjected to the stress concentration, as shown by FEA (Fig. 2), the higher modulus and lower toughness of enamel cannot adequately transfer the stress from the neck region to the test region without failure.

Stress transference from the gripping region to the testing region (gauge) will improve directly with the notch radius of curvature, also referred to as the 'neck'. Teeth; however, physically put limits on overall specimen geometry. The observance of failures within the gauge section verifies, following St. Venant's principle [69,70], that the notching was located far enough away from the middle of the specimen for resin–dentin specimens. The bond line located anywhere within the gauge section, theoretically, permits a suitable test, however, the technical ability to place the bond line in the gauge section during trimming also limits gauge section length.

Due to lack of consensus of specimen design and the relatively higher incidence of PTFs during trimming, some laboratories have recommended the notchless stick or 'non-trimming' technique [16,45]. Stick-shaped specimens are simple to prepare and when compared to a dumbbell geometry with a rectangular testing region had similar bond strengths, stress concentrations and failure locations; whereas an hourglass geometry was found to be significantly different to the stick and dumbbell geometries and more sensitive to flaws introduced during specimen preparation [39,71]. Sadek et al., demonstrated that the use of the diamond saw produces defects on corners of the sticks, resulting in high levels of PTFs (35.4% for enamel and 18.2% for dentin) which could be

Table 3 – Enamel and dentin 24 h μTBS using five different bonding agents using the methods described in Sattabanasuk et al. [60] (unpublished data).

	Bonding agents				
	A	B	C	D	E
Unground enamel[a]					
Mean bond strength with PTFs[b] (MPa)	31.0	5.3	41.9	34.1	11.9
[Coefficient of variation %]	[34]	[158]	[38]	[42]	[121]
Pre-test failures	1/20	15/20	0/20	1/20	11/20
Bond strength excluding PTFs (MPa)	32.6	18.1	41.9	35.9	25.3
[Coefficient of variation]	[24]	[41]	[38]	[35]	[45]
Non-gauge fracture[c]	4/20	0/20	5/20	2/20	2/20
Ground enamel					
Mean bond strength with PTFs[b] (MPa)	33.8	3.7	40.8	37.7	28.2
[Coefficient of variation %]	[31]	[232]	[44]	[26]	[55]
Pre-test failures	0/20	18/20	0/20	0/20	4/20
Bond strength excluding PTFs (MPa)	33.8	28.4	40.8	37.7	35.0
[Coefficient of variation]	[31]	[31]	[44]	[26]	[22]
Non-gauge fracture[c]	5/20	0/20	9/20	12/20	0/20
Dentin					
Mean bond strength with PTFs[b] (MPa)	58.2	38.3	56.4	55.9	55.0
[Coefficient of variation %]	[18]	[46]	[16]	[23]	[21]
Pre-test failures	0/30	2/30	0/30	0/30	0/30
Bond strength excluding PTFs (MPa)	58.2	41.0	56.4	55.9	55.0
[Coefficient of variation]	[18]	[36]	[16]	[23]	[21]
Non-gauge fracture[c]	2/30	1/30	1/30	3/30	3/30

[a] Surface area of unground enamel assumed to be equal to flat ground enamel and dentin.
[b] PTFs included as 1 MPa.
[c] All non-gauge fractures involved a portion of the gauge length in debond pathway.

reduced by diamond wire sectioning [72]. The relatively high incidence of PTFs observed can be explained by the stress concentrations generated by the specimen preparation defects at the sharp corners and by the material property differences of the substrates. This can be improved by using a round cross-sectional testing region or maybe by chamfering the corners of square and rectangular specimens. The testing region geometry is rarely reported or discussed when a particular specimen geometry is recommended as being most appropriate for μTBS testing. Although producing a more favorable stress distribution, fabricating a μTBS dumbbell-shaped specimen with a round testing region requires special equipment or computer-numerically controlled fabrication methods that are currently cost-prohibitive for wide adoption [38].

Trimming is very technique sensitive and it induces additional stress as reflected in the number of specimens that fail prior to testing, especially in weaker bonds or specimens with relatively brittle behavior [42,66]. The operator's experience and manual skill will, therefore, influence the results and the quality of the study [36]. It is difficult to standardize freehand trimmed specimens to consistently produce the test region of the bonded interface, the results of which, can significantly affect the stress distribution [38,68]. In addition, the action of the bur can be particularly aggressive as uneven cutting forces may be applied.

As years of research and clinical experience have clearly demonstrated, adhesion to acid-etched enamel is more reliable than to dentin. However, owing to the intrinsic brittleness of this tissue relative to dentin, it will fail under relatively lower loading levels with the surface areas used in μTBS and with greater variability (Table 3) [73]. Microtensile testing has been questioned as an appropriate trial for enamel [74], which is fragile, anisotropic, and has water content lower than dentin. Pre-testing microscopic analysis of prepared sticks revealed a more frequent occurrence of microcracks in enamel than in dentin, with microcracks most often located at the periphery of the sticks, suggesting flaw introduction during preparation [72].

The thickness (<1.5 mm) of the specimen affects its ability to survive the preparation procedures necessary for microtensile testing. A relatively high incidence of PTFs (26%) during hourglass trimming of microtensile dentin specimens to create a 0.5 mm wide bonding has been reported [45]. Phrukkanon et al., recommended never reducing the cross-sectional area at the bonding interface to less than 1.1 mm^2 due to an observed high pre-test failure rate [18]. This calls into question the definition and utility of the term "microtensile". Shono et al., related the rate of PTFs using a trimming technique to the mean tensile bond strength measured and reported that groups with bond strengths 13 MPa or lower were less likely to survive preparation [11]. The non-trimming technique imparts less cumulative damage to the specimen since bond strengths as low as 5 MPa have been measured [16]. However, the non-trimming technique does not create a defined test region with uniform stress state.

4.3. *Test speed*

A human chewing-cycle lasts around 800 ms, with the closing movement reaching duration of less than 400 ms [75]. This translates into over 2000 mm/s, a value 500 times higher than what may be employed in conventional bond strength test-

ing [76]. However, such high rates are limited by the effective loading rate of testing equipment. Few *in vitro* studies have been carried out to address the influence of crosshead speed on μTBS [42,77,78]. These studies unanimously reported that there was no difference on microtensile bond strength in the range of crosshead speeds evaluated (0.01–10.0 mm/min). According to Yamaguchi et al., the smaller specimens used in microtensile testing contain a lower number of internal defects and a more homogeneous distribution relative to "macro" testing; therefore, such samples exhibit independence of strain-rate which reduces the impact of speed on bond strength values [78]. However, the time–temperature equivalence of viscoelastic materials tell us that at very low or high testing speeds nominal bond strengths should reflect a strain-rate sensitivity. Poitevin et al., reported that the lower the speed, the greater the difference between stress at maximum load and stress at breaking; regardless, they recommended 1 mm/min crosshead speed because of a more uniform stress-time pattern [42].

4.4. *Role of FEA in the interpretation of "micro" bond strength testing*

Finite element analysis is a very powerful tool that can assist in our understanding of bond strength testing parameters and the interpretation of test results. Studies using FEA have been referenced throughout this review with little explanation due to space limitations. The nominal μTBS is not determined solely by adhesive bond properties. The initiation of fracture during testing is due to the critical combination of stress and defect size [24,79]. Defect size is, at least theoretically, controllable and verifiable; however, stress distribution is determined by mechanical properties of components, shape of the specimen, and the load magnitude and direction. The advantage of using FEA is that it allows separation of these parameters and their effects [38,39]. The combination of diverse materials and complex geometry makes stress distribution analysis in teeth very complicated [37]. Nevertheless, FEA of stress distribution has been used to study the sensitivity of bond strengths to specimen design, imperfections introduced during specimen fabrication, and changes in testing conditions [38,39,71]. Stress distribution in different experimental conditions can then be qualitatively related to the most probable sites of failure initiation [80]. This possibility does not exist within mechanical tests alone. Some studies have used FEA only to predict the parameters of the experimental conditions [38]; however, numerical models require experimental validation and FEA results that have not been laboratory validated should be viewed with reservation [37,39]. On the other hand, experimental methods that do not take into consideration mechanical properties of the materials used and the experimental test conditions should also be analyzed prudently. Fractographic microscopy of debonded specimens is also highly encouraged for clarification of FEA predictions and mechanical test results.

Numerical analyses using FEA can satisfactorily determine states of stress and strain and energy release rates or stress-intensity factors within a bonded joint [81]. However, convergence of stress and strain produced by the finite element codes in the vicinity of boundary corners or material discontinuities, even with good mesh refinement, can occur. A critical consideration is the parameters chosen to analyze the FEA outcomes. Frequently von Mises criterion is used, without consideration of the type of the stress concentration, i.e., compressive or tensile. The Maximum Principal Stress is proposed as a more appropriate parameter to analyze μTBS testing because the maximum tensile stresses are shown isolated, and the stress concentration factor can be calculated as K_t, that is, ratio of maximum principal and nominal stresses. However, if only a punctual stress is recorded, this value may be influenced by mesh quality and boundary conditions. Therefore, it is recommended to use the top 5% of test values to calculate quantitatively the Maximum Principal Stress parameters. Indeed the equivalent stress can also be applied using the subroutine, when taking into account that dental structures and most dental materials, being relatively brittle, fail more easily under tensile loads. The equivalent stress combines the six stress components from the three-dimensional stress state according to a relationship known as the von Mises criterion [82]. Since dentin strength in tension is approximately three times lower than in compression [83], the von Mises criterion can be modified to account for the fact that tensile stress components are more critical than compressive [32], resulting in a more adequate analytical parameter, called, Modified von Mises equivalent stress.

5. Summary

Before bond strength testing can be standardized we must understand how the measured nominal bond strength is related to the local stress distributions generated during testing and, most importantly, how this information relates to clinical performance. As stated by Sudsangiam and van Noort, "Inherent in the process of standardization is the belief that the results derived from the bond strength test will have some validity and meaning as long as bond strength can be measured consistently. This is highly questionable ... because no amount of standardization will overcome inconsistency problems if a test is fundamentally flawed" [84]. Until such time that the relationship between a particular bond strength test and clinical performance is fully understood, more pragmatic goals may be the following: (1) adoption of universally accepted terminology and definitions, (2) standardized reporting of specimen handling and fabrication, (3) inclusion of positive and negative controls during testing, (4) standardized reporting of experimental set-up and test mechanics, and (5) full reporting of, or access to, a complete data set.

Thorough documentation and communication of how bond strength tests, whether "micro" or "macro", are conducted will assist greatly in furthering our understanding of the strengths and limitations of various testing methods and should help narrow the knowledge gap between the laboratory and clinic (Appendix A). Stick-shaped specimens glued by all gripping surfaces and passively gripped dumbbell-shaped specimens with round cross-sectional gauge lengths hold much promise, but more studies remain to be done before conclusions can be drawn. Bond strength tests, regardless of type and size, remain useful as screening tools for new

adhesive approaches and investigation of experimental variables. These remain strength-based testing approaches of a complicated adhesive joint with all inherent limitations [31], therefore, if ones goal is to measure an adhesive bond material property, a fracture mechanics approach, not without great difficulty, may prove more successful.

Appendix A. Appendix[3]

- "Uniaxially loaded tensile strength tests provide information on strength-limiting flaws from a greater volume of uniformly stressed material. Therefore, because of the probabilistic strength distributions of ceramics (adhesive resin–tooth structure bonds), a sufficient number of specimens at each testing condition is required for statistical analysis" (in contrast to a sufficient number of measurements to account for any component of uncertainty arising from a systematic effect or error).
- "Strengths obtained using different volumes or surface areas of material in the gauge sections will be different due to these sizes, ... resulting strength values can be scaled to an effective volume or surface area of unity."
- "Results of tensile tests of specimens fabricated to standardized dimensions from a particular sized material and/or selected portions of a part may not totally represent the strength and deformation properties of the entire full-size end product or its in-service behavior in different environments."
- "Surface preparation of test specimens can introduce fabrication flaws that may have pronounced effects on tensile strength. Universal or standardized test methods of surface preparations do not exist. It should be understood that final machining steps may or may not negate machining damage introduced during the early coarse or intermediate machining. Thus specimen fabrication history may play an important role in the measured strength distributions and should be reported."
- "Bending in uniaxial tensile tests can cause or promote non-uniform stress distributions with maximum stresses occurring at the specimen surface leading to non-representative fractures originating at surfaces or near geometrical transitions. ... Similarly, fracture from surface flaws may be accentuated or muted by the presence of the non-uniform stresses."
- "The brittle nature of advanced ceramics (enamel, dentin, RBC behaves in a brittle manner also) requires a uniform interface between the grip components and the gripped section of the specimen."
- "Gripping devices can be classed generally as those employing active (e.g., glue) and those employing passive (e.g., Dircks device) grip interfaces.
  - The important aspect of passive grip interfaces is uniform contact between the gripped section of the specimen and the grip faces.
  - Moderately close tolerances are required for concentricity of both the grip and specimen diameters."
- "Regardless of which type of coupler is used, alignment of the testing system must be verified at the beginning and end of a test series."
- "Allowable Bending—analytical and empirical studies have concluded that for negligible effects on the estimates of strength distribution parameters (for example, Weibull modulus, m, and characteristic strength, σ_φ) allowable percent bending should not exceed five."
- "At a minimum, an autographic record of applied load *vs.* time should be obtained. Crosshead displacement of the test machine may also be recorded but should not be used to define displacement or strain in the gauge section especially when self-aligning couplers are used in the load train."
- "Flat tensile specimens—... Disadvantages include the relatively small volume of material tested and the sensitivity of the specimen to small dimensional tolerances or disturbances in the load train. It should be noted that the gauge section of flat tensile specimens for tensile strength measurements are sometimes cylindrical. While this type of gauge section adds to the difficulty of fabrication and therefore the cost of the flat specimen if does avoid the problem of fractures initiating at corners of non-cylindrical gauge sections. Corner fractures may be initiated by stress concentrations due to the elastic constraint of the corners but are more generally initiated by damage (chipping, etc.) that can be treated by chamfering the corners ..."
- "Final finishing should be performed with diamond tools that have between 320 and 600 grit (35–14 μm). No less than 0.06 mm per face should be removed during the final finishing phase, and at a rate not more than 0.002 mm per pass."
- "Generally, surface finishes on the order of average roughnesses, Ra, of 0.2–0.4 μm is recommended to minimize surface fractures related to surface roughness. However, in some cases the final surface finish may not be as important as the route of fabrication due to the generation of subsurface damage during the fabrication process."
- "In many instances, the bulk of the material is removed in a circumferential grinding operation with a final, longitudinal grinding operation performed in the gauge section to assure that any residual grinding marks are parallel to the applied stress."
- "Generally, computer numerically controlled (CNC) fabrication methods are necessary to obtain consistent specimens with the proper dimensions within the required tolerances. A necessary condition for this consistency is the complete fabrication of the specimen without removing it from the grinding apparatus."
- "Number of test specimens—use C1239 Practice for reporting uniaxial strength data and estimating Weibull parameters for advanced ceramics."

[3] The following guidelines are from ASTM C 1273: standard test method for tensile strength of monolithic advanced ceramics at ambient temperatures. Philadelphia, PA, 1995 (with comments added). Although this standard is not written for tensile testing of two substrates joined by an adhesive bond, the writers contend that the statements included below are applicable to the adhesive resin–tooth bond due to the inherently brittle behavior of all components of the bonded assemble and for reasons stated previously in the body of the paper.

- "Load rate—for most advanced ceramics exhibiting linear elastic behavior, fracture is attributed to a weakest-link fracture mechanism generally attributed to stress-controlled fracture from Griffith-like flaws. …. Alternatively, select stress rates to produce final fracture in 5–10 s to minimize environmental effects when testing in ambient air."
- "Note that results from specimens fracturing outside the uniformly stressed gauge section are not recommended for use in the direct calculation of a mean tensile strength. Results from specimens fracturing outside the gauge section are considered anomalous and can be used only as censored [right] tests, i.e., specimens in which a tensile stress at least equal to that calculated" by fractures occurring in the "uniform gauge section before the test was prematurely terminated by a non-gauge section fracture."
- "Fractographic examination of each failed specimen is highly recommended to characterize the fracture origins."
- Test report should include:
  - Tensile test geometry (include engineering drawing).
  - Type and configuration of the test machine.
  - Type and configuration of grip interface used (include drawing as needed).
  - Type and configuration of load train couplers (include drawing as needed).
  - Number of specimens tested validly (that is, fracture in the gauge section). In addition report total of number of specimens tested to provide an indication of the expected success rate of the particular specimen geometry and test apparatus.
  - All relevant material data (tooth substrate description, restorative materials, etc.).
  - Description of the method of specimen preparation including all stages of machining.
  - Testing environment: temperature and humidity.
  - Test mode (load, displacement, or strain control).
  - Test rate (load rate, displacement rate, or strain-rate.
  - Percent bending and corresponding average percent strain.
  - Mean tensile strength, standard deviation and coefficient of variation.
  - Estimates of strength distribution parameters (for example, Weibull modulus and characteristic strength).
  - Pertinent overall specimen dimensions.
  - Average surface roughness.
  - Average cross-sectional dimensions.
  - Pre-load rate and value.
  - Fracture location relative to the gauge section midpoint.
  - Type and location of fracture origin (flaw) relative to the front of the specimen as marked.

Added suggestions:

- Describe specimen fabrication and geometry in sufficient detail, to include length and shape of gauge region, such as, cylindrical, square, square with chamfered corners.
- Report all modes of failure (locus in the adhesive bond through which the failure propagates) with consistent nomenclature and definition.
  Cohesive in tooth structure adherend (enamel, dentin, etc.).
  Cohesive in restorative material adherend (RBC, RMGI, etc.).
  Adhesive joint or adhesive bond: defined from interface of RBC and adhesive resin to the bottom of the hybrid layer (RBC–AR to BHL–dentin). Pocious recommends using the phrase "apparent" failure in adhesive joint or bond since sophisticated surface chemistry analysis is required to confirm a true "adhesive failure" between the adhesive system and the adherend.
  Mixed failure: in adhesive joint and adherend(s).
- Encourage identification, if possible, of fracture origin. The fracture origin is the flaw from which the strength-limiting crack began to produce the debond pathway or mode of failure. The debond pathway can be relatively easily identified when using adequate magnification; however, the true fracture "origin" requires fractographic expertise and, due to the viscoelastic nature of materials in the adhesive resin–tooth structure bonded joint, may be very difficult to precisely identify.
- Report and include all pre-test specimen failures as left-censored data with a defined value.
- Include non-gauge section or test region (if no gauge region exists) failures as right-censored data.
- Report descriptive and inferential results using both normal and Weibull distribution statistics.
- Use random effects (ANOVA) and frailty effects (Weibull) for clustered data, i.e., multiply specimens from the same tooth. This accounts for tooth dependency while using specimen as the experimental unit.
- To reduce material property mismatch across the adhesive joint, consider the use of a single adherend, such as, dentin bonded to dentin, RBC bonded to RBC.
- Additional terminology:

"Stick": square or nearly square microtensile specimen (sometimes referred to as a "beam").

"Slab": rectangular microtensile specimen.

"Notch": any attempt at narrowing the specimen to concentrate stress in a test region.

"Dumbbell": a notched specimen with a defined radius of curvature from the end or shoulder of the specimen to a straight gauge length that includes the adhesive joint.

"Hourglass": a notched specimen with a defined radius of curvature from the end or shoulder of the specimen to the adhesive joint without a defined gauge length.

"Active gripping method": mechanical fastening of specimen to gripping device, such as glue or clamps.

"Passive gripping method": specimen is placed in a testing device without the aid of glue or mechanical gripping; device should self-align the specimen parallel to the tensile load.

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