The validity of reported tensile bond strength utilizing non-standardized specimen surface areas. An analysis of *in vitro* studies

CATHERINE STAMATACOS-MERCER, DDS & TIMOTHY L. HOTTEL, DDS, MS, MBA

ABSTRACT: ***Purpose***: To assess and discuss the true value and perceived relevance of present day dental adhesive tensile bond strength studies. There are flaws and inconsistencies present in the data due to the inherent variations in testing methods, conditions, and types of samples prepared. In particular, surface areas of specimens need to be standardized. This review considers the significant impact of different surface areas of tooth specimens utilized in testing. ***Results:*** On review of the data, relatively higher MPa values do not necessarily indicate improved dental adhesive products or procedures (*Am J Dent* 2005;18:105-108).

CLINICAL SIGNIFICANCE: Present day data reporting high dentin bond strengths utilizing specimen sizes that are not standardized may be misinterpreted to mean better clinical products and clinical results.

⌧: Dr. C. Stamatacos-Mercer, Department of Restorative Dentistry, College of Dental Medicine, Nova Southeastern University, Fort Lauderdale, FL, USA. E-⌧: stamatac@nsu.nova.edu

Introduction

Evaluation of dental adhesives is controversial. There are inherent fundamental flaws and limitations to the data using today's methods of testing which result in inconsistent results. Adhesion analysis of dentin bonding agents has been performed by numerous mechanical testing methods including shear bond strength, tensile bond strength (TBS), microleakage and contraction gap size measurements. Dental manufacturers rely largely on *in vitro* testing to predict the clinical performance of a product.[1] They predominantly utilize tensile bond strength studies to evaluate and market their products. However, *in vitro* bond strength assessments have been poor indicators of clinical success.[2,3] Even if reported high tensile bond strengths reflect a stronger bond to dentin, there is no statistical correlation between that and microleakage[4,5] or resin penetration into dentin.[6] Therefore, bond strength testing is only one parameter to help predict clinical performance.

The differences in bond strength results are probably due to variations in the quality of the dentin and in tensile test methods.[7] The variations in bond strengths are usually considered to be related to the different adhesive procedures, however far too little attention has been paid to the details of the testing conditions[8] and the critical role of specimen size.

The tensile bond strength (TBS) test is very sensitive, therefore understanding test methodology and resultant interpretation of the data is extremely critical. Bond strength values for identical products obtained from conventional tensile and microtensile testing can vary greatly depending on the condition and size of specimens. Sano *et al*[9] demonstrated that tensile strength is inversely proportional to the bonded surface area. The resultant higher tensile strengths reported after the development of the microtensile test method by Sano *et al*[9] are actually a necessary end result of significantly smaller and smaller specimen surface areas being utilized in microtensile test procedures. Not enough attention has been paid to the possibility that the recent high values of tensile bond strengths reported in the literature may not necessarily mean that the adhesive systems being tested are significantly stronger than those of the past.

The International Organization for Standardization gives researchers guidelines for standardization of variables and therefore improvement in the consistency of tensile bond strength methodologies. Although many variables for TBS study methodology are standardized, dimensions for a standardized surface area do not exist. The ability to reproduce data between laboratories has been limited due to the lack of experimental standardization and variability of substrate.[11-13] The lack of consistent values for dentin bond strengths in shear or tension, from what are superficially identical experimental procedures, has led to ambiguities in the interpretation of the data.[8] Small alterations in the specimen or in the stress distribution during load application have a great influence on the result.[9,14] Van Noort *et al*[8] concluded that bond strength tests between dental materials and tooth tissues are so severely affected by the test conditions that the comparison of data from different laboratories is probably impossible. Even for similar tests performed in the same laboratory, large variations can arise.[8] Table 1 lists the mean TBS results of various investigations cited in this paper and shows the vast differences in MPa ranges. Note the differences in results between conventional and microtensile bond strength studies and how surface area affects the results of these tests for reasons discussed in the Introduction (Table1).

The publication of dentin bonding strength data is prolific in the dental literature and will remain so as more and more new dentin bonding agents are marketed. It is the purpose of this presentation to assess the usefulness of this information due to inherent inconsistencies and to question the perceived relevance of these bond strength studies. The often overlooked role of specimen size, which is critical to the interpretation of tensile strength studies, is discussed.

Discussion

Differences in reported bond strength data can be attributed, in many instances, to a lack of adequate control of the variables in the test procedures utilized. This is attributed not only to the differences in the properties of the adhesive materials and the characteristics of dentin, but also to the dif-

Table 1. Results of tensile bond strength studies.

Product	Type of TBS	Surface area or diameter	Mean MPa	SD	Study	Year
Prime&Bond 2.1[c]	Microtensile	N/A	54.30	15.6	Cardoso[36]	2001
Prime&Bond 2.0[c]	Tensile	88.8 mm^2 SA	3.96	1.24	Del Nero[37]	1999
Prime&Bond NT[c]	Microtensile	N/A	19.0	3.90	Frankenberger[38]	1999
Prime&Bond NT	Microtensile	N/A	62.0	17.60	Cardoso[36]	2001
Prime&Bond NT	Microtensile	0.35-0.45 mmcs	48.2	N/A	Nunes[39]	2001
Prime&Bond NT/NRC[c]	Microtensile	1.2 mm d	29.9	N/A	Tanumiharja[15]	2000
Scothchbond MP	Tensile	3.0 mm d	9.65	4.78	Cardoso[21]	1998
Scothchbond MP	Microtensile	0.25 mm^{2}csSA	32.74	12.52	Cardoso[21]	1998
Scothchbond MP	Microtensile	1-2 mm cs	20.3	5.5	Nakajima[29]	1995
Scothchbond MP	Microtensile	<0.4 mm^2 SA	38.0	N/A	Sano[9]	1994
Scothchbond MP	Tensile	4x7 mm rect.	2.68	1.85	Tam[40]	1993
Scothchbond MP	Tensile	4x7 mm rect.	4.88	1.88	Tam[40]	1993
Scotchbond 2[a]	Tensile	4.0 mm d	3.10	1.99	Van Noort[14]	1991
Scotchbond 2	Tensile	4.0 mm d (w/flash)	6.9	2.05	Van Noort[14]	1991
Scotchbond 2	Tensile	4x7 mm rect	1.72	0.90	Tam[40]	1993
Scotchbond 2	Tensile	4x7 mm rect	1.63	1.07	Tam[40]	1993
Scotchbond 2	Tensile	5.0 mm d	7.7	2.8	Perinka[20]	1992
Scotchbond 2	Tensile	5.0 mm d	3.5	N/A	Øilo[7]	1990
Scotchbond 2	Tensile	3.0 mm d	15.2	N/A	Øilo [7]	1990
Scotchbond (dual)	Tensile	5.0 mm d	1.4	N/A	Øilo[7]	1990
Single Bond	Tensile	3.0 mm	9.34	4.33	Cardoso[21]	1998
Single Bond	Microtensile	0.25 mm^2	34.60	10.88	Cardoso[21]	1998
Single Bond	Tensile	3.0 mm d	18.1	2.4	Spohr[41]	2001
All Bond 2[d]	Microtensile	0.5 mm^{2}SA	40.7	9.0	Armstrong[42]	1998
All Bond 2	Microtensile	1-2 mm crs.	26.9	8.8	Nakajima[29]	1995
All Bond 2	Tensile	4 x 7 mm rect.	8.39	3.99	Tam[41]	1993
All Bond 2	Tensile	4 x 7 mm rect.	6.39	2.55	Tam[41]	1993
Clearfil Liner Bond 2[e]	Microtensile	1-2 mm crs	29.5	10.9	Nakajima[29]	1995
Clearfil Liner Bond 2	Microtensile	>0.4 mm^2 SA	55.0	N/A	Sano[9]	1994
Clearfil Liner Bond 2	Tensile	3.0 mm d	24.0	3.3	Spohr[41]	2001
Clearfil Liner Bond 2	Microtensile	1.0 mm^2 SA	28.9	5.2	Yoshiyama[43]	1998
Clearfil Liner Bond 2	Microtensile	1.2 mm d	36.0	N/A	Tanumiharja[15]	2000
Etch and Prime 3.0	Tensile	3.0 mm	6.43	2.81	Cardoso[21]	1998
Etch and Prime 3.0	Microtensile	0.25 mm^2	27.77	7.88	Cardoso[21]	1998
Etch and Prime 3.0	Tensile	3.0 mm d	5.8	2.4	Spohr[41]	2001
Gluma[e]	Tensile	5.0 mm d	3.5	N/A	Øilo[7]	1990
Gluma	Tensile	3.0 mm d	12.0	N/A	Øilo[7]	1990

SD= standard deviation; d= diameter; SA= surface area; cs= cross section; crs= cross sectional area; csSA= cross sectional surface area; rect.= rectangle; UP= unpublished data; N/A= Not available.

ferences in specimen preparation, size and test methodology.[15] Therefore, variations and the resultant data interpretation of tensile bond strength studies must be carefully analyzed. For a given bonding agent, bond strengths vary greatly, not only among different studies, but also within studies.[16,17] In particular, the role of the size of the specimens used and the resultant surface area tested is highly significant. According to Griffith's defect theory,[18] when testing uniform materials in tension, the tensile strength of a material decreases with increasing specimen size. Sano *et al*[9] demonstrated that tensile strength is inversely proportional to the bonded surface area. This means that smaller surface areas are associated with higher tensile bond strengths, and conversely, larger surface areas are associated with lower tensile bond strengths. The reasons for this phenomenon may be due to the effect of the presence of defects and/or stress raisers at the interface or in the substrate. Larger specimens probably contain many more defects compared to smaller specimens.[9]

A critical point in data interpretation needs to be addressed. Not enough emphasis is placed on the role of surface area and its dramatic effect on MPa values. In the Øilo & Olsson[7] study, the bond strengths were found to be dependent upon the tensile test method used. In conventional tensile bond studies, specimen surface areas tested are much larger than the surface areas tested in the microtensile bond strength test. A microtensile method to evaluate specimens with small surface areas (ca. 1 mm^2), was developed by Sano *et al.*[9] Therefore, the MPa bond strength numbers found in microtensile bond strength studies can be more than double those of conventional tensile bond strength studies.[1,19] It is important to distinguish between the two types of tests and the variances in the results, which are directly related to the surface area of the samples.

Great differences in values in bond strength studies are directly related to whether microtensile or conventional methodologies are utilized and do not necessarily indicate that the products themselves have improved dramatically. The higher bond strengths that are obtained with the microtensile bond method are presumably due to better stress distribution during testing[20] and do not represent improved products or bonding procedures. Using this method often results in higher apparent bond strengths at failure than are found in larger

Table 2. Cardoso[21] research data, 1998.

Product	Type of TBS test	Mean MPa
Scotchbond MP	Tensile	9.65 ± 4.78
Scotchbond MP	Microtensile	32.74 ± 12.52
Etch & Prime 3.0	Tensile	6.43 ± 2.81
Etch & Prime 3.0	Microtensile	27.77 ± 7.88
Single Bond	Tensile	9.34 ± 4.33
Single Bond	Microtensile	34.60 ± 10.88

samples.[21] Even though the same product is being tested, the reported MPa may be vastly different. Even standardizations of bond strength studies do not overcome the problem of data interpretation. For example, Cardoso *et al*[21] tested the products SingleBond,[a] Etch&Prime 3.0[b] and Scothchbond MP.[a] Both microtensile and conventional tensile testing were used in this study. Of all the studies, this one should have the least variables in comparing test results because the two types of testing were performed in the same laboratory. The microtensile test values were 3 to 4 times greater than the conventional tensile test for the same products. This supports the claim that the dentin bonding agents have not really improved as much as manufacturers suggest and that it is the testing methodology and variables (particularly sample size) that affect the MPa results (Table 2).

Surface areas of specimens need to be standardized. Manufacturers that use the microtensile test method and report higher MPa values may lead the average practitioner to believe that their product is superior. This is probably not the case. Published data[22-27] shows that the nominal tensile bond strength for dental systems varies from 2 to 6 MPa for bonds between composite resin and dentin when using a dentin bonding agent.

Today the average practitioner reads a study where higher and higher *in vitro* bond strengths are reported. But are these results actually a true indication of higher bond strengths? The authors submit that if microtensile testing were used in the 1980's, MPa values of products at that time would have to have been much higher. The fact that they would show higher MPa values would not indicate better adhesive properties.

Recently, the consensus[28] is that the microtensile bond strength studies are more desirable and have the following advantages: (1) More adhesive fractures, fewer cohesive fractures; (cohesive fractures are supposedly not desirable because they do not test the true interfacial bond strength.) (2) higher interfacial bond strengths can be measured; (3) ability to measure regional bond strengths; (4) means and variances can be calculated for single teeth; (5) permits testing of bonds to irregular surfaces; (6) permits testing of very small surface areas; and (7) facilitates examination of the failed bonds by SEM. Also, this testing method may create a more uniform stress distribution compared with the conventional method.[29] This method does measure the interfacial bond strength more precisely in a significantly smaller sample, helps obtain true ultimate stress and delivers a more uniform stress, but is this relevant *in vivo*? Clinically, restorations have a very large surface area and are placed into a three-dimensional cavity preparation. Much greater stresses occur within the material in a three-dimensional cavity preparation.[30,31] Resin-based composites shrink as they polymerize and contraction stresses of up to 7 MPa develop within the resin.[30,32,33] But when resin is bonded to a single surface, as they are for bond strength studies, flow relaxation occurs relieving some of the contraction forces and therefore, these values are not realistic. It was concluded by Van Noort *et al*[8] that the actual stresses have little relationship to the average stress values determined by bond strength testing.

The conventional test method possibly places a more realistic MPa value on adhesives used today. Although three-dimensionality is not addressed, utilizing a larger surface area could be a more realistic representation of how these resins present *in vivo* and therefore a product that showed an increase in bond strength using the conventional method may be more significant. Although this method is not a way to obtain true ultimate strength of an adhesive, the larger surface areas used in this method are more representative of what occurs clinically. Non-uniform stresses would seem more relevant during a test because occlusal forces *in vivo* are not uniform. Conventional testing causes more non-uniform stresses and the small defects and voids in a larger test sample are more characteristic of an *in vivo* restoration.

Conventional testing served relatively well when resin-dentin bond strengths were relatively low.[34] When bond strengths improved, with the advent of new products, cohesive failures appeared. These failures, which leave the resin-dentin interface intact, were not desirable because such failures preclude measurement of interfacial bond strengths and limit further improvements.[34] Perhaps these TBS results associated with cohesive failures could relate more to the strength of the dentin and the area of dentin fracture than to the strength of the actual bonded interface.[35]

Clinical consequences - The much higher microtensile bond strength values that are being reported in the literature may be giving the average practicing dentist a false sense of security regarding the "real" improvement of dentin adhesive systems. The lack of data interpretation concerning the much smaller sample sizes and the inherent higher MPa values associated with microtensile bond strength studies needs to be carefully examined. Practitioners need to understand that the variations and inconsistencies associated with these studies may lead to misinformation concerning the clinical performance of the products being tested.

There is still a real need for re-evaluation and standardization of test procedures to help clinicians assess the true effectiveness of these dentin adhesive products. Even if standardization is achieved, bond strength studies can only be used for a comparison of the effectiveness of the bonding agents and cannot be related directly to what might happen clinically.

This review hopes to show that although the microtensile method certainly has its value, too much emphasis is placed on the reported high values and that the corresponding interpretation of better clinical performance may be exaggerated. The much higher tensile bond strength MPa results that have been published in recent years can be misinterpreted and misleading. Dentin adhesive products have probably not improved as much as manufacturers of these products seem to claim. Recent products which have been advertised as having high MPa values using microtensile methods would necessarily exhibit much lower tensile bond strengths if conventional methods were utilized. The clinician may not be aware that the different surface areas utilized significantly impacts the bond strength values reported. The interpretation of the many marketing claims and published bond strengths may not have as much significance in determining whether a product will be successful clinically as manufacturers would like practitioners to believe. This review hopes to lead to the exploration of alternative approaches of testing and to the standardization of specimen surface areas utilized in tensile bond strength studies.

The following would improve the validity of dental adhesive evaluations:

- A universal sample size needs to be established for future tensile bond strength studies.
- Existing variables that are standardized by the International Organization for Standardization should be adhered to by researchers.
- Products should undergo a combination of testing. Improvement in standardization of tensile bond strength study variables (as well as shear bond strength study variables) should be combined with standardized data from gap measurement tests, microleakage tests and the clinical usage tests for a more realistic correlation between laboratory results and clinical performance.

a. 3M ESPE, St. Paul MN, USA.
b. Degussa AG, Geschaftsbereich Dental, Hanau, Germany.
c. Dentsply Caulk, Milford, DE, USA.
d. Bisco, Schaumburg, IL, USA.
e. Kuraray Co., Ltd., Osaka, Japan.

Dr. Stamatacos-Mercer is Assistant Professor, Department of Restorative Dentistry, and Dr. Hottel is Professor, Department of Prosthodontics, Executive Associate Dean and Associate Dean for Academic and Financial Affairs, Nova Southeastern University, Fort Lauderdale, Florida, USA.

References

1. Swift EJ Jr., Perdigão J, Heymann H. Bonding to enamel and dentin: A brief history and state of the art. *Quintessence Int* 1995; 26: 95-110.
2. Tyas MJ. Guest editorial: Reliability and validity in dental materials testing. *J Dent Res* 1991; 70: 1471.
3. De Hoff PH, Anusavice KJ, Wang Z. Three-dimensional finite element analysis of the shear bond strength. *Dent Mater* 1995; 11: 126-131.
4. Fortin D, Swift E Jr., Denehy G, Reinhart JW. Bond strength and microleakage of current dentin adhesives. *Dent Mater* 1994; 10: 253-268.
5. Holderegger C, Paul SJ, Luthy H, Scharer P. Bond strength of one-bottle dentin bonding agents on human dentin. *Am J Dent* 1997; 10: 71-75.
6. Finger W, Fritz U. Laboratory evaluation of one-component enamel/dentin bonding agents. *Am J* Dent 1996; 9: 206-210.
7. Øilo G, Olsson S. Tensile bond strength of dentin adhesives: A comparison of materials and methods. *Dent Mater* 1990; 6: 138-144.
8. Van Noort R, Noroozi S, Howard IC, Cardew GE. A critique of bond strength measurement. *J Dent* 1989; 17: 61-67.
9. Sano H, Sonoda H, Shono T, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength. Evaluation of a micro tensile bond test. *Dent Mater* 1994; 10: 236-240.
10. International Organization of Standardization, [ISO/TS 11405:2003(E)].
11. Retief DH. Standardizing laboratory adhesion tests. *Am J Dent* 1991; 4: 231-236.
12. Söderholm KJ. Correlation of *in vivo* and *in vitro* performance of adhesive resto-rative materials: A report of the ASC MD156 Task Group on test methods for the adhesion of restorative material. *Dent Mater* 1991; 7: 74-83.
13. Fowler C, Swartz M, Moore B, Rodes B. Influence of selected variables in adhesion testing. *Dent Mater* 1992; 8: 265-269.
14. Van Noort R, Cardew GE, Howard IC, Noroozi S. The effect of local interfacial geometry on the measurement of the tensile bond strength to dentin. *J Dent Res* 1991; 70: 889-893.
15. Tanumiharja M, Burrow MF, Tyas MJ. Microtensile bond strengths of seven dentin adhesive systems. *Dent Mater* 2000; 16: 180-187.
16. Prati C, Biagini G, Rizzoli C, Nucci C, Zucchini C, Montanari G. Shear bond strength and SEM evaluation of dentin bonding systems. *Am J Dent* 1990; 3: 283-288.
17. Retief DH, O'Brian JA, Smith LA, Marchman JL. *In vitro* investigation and evaluation of dentin bonding agents. *Am J Dent* 1988: 1: 176-183.
18. Griffith AA. The phenomena of rupture and flow in solids. *Philo Trans R Soc Lond* 1920: A221: 168- 198.
19. Sano H, Ciucchi B, Matthews WG, Pashley DH. Tensile properties of mineralized human and bovine dentin. *Oper Dent* 1994; 10: 236-240.
20. Perinka L, Sano H, Hosoda H. Dentin thickness, hardness, and Ca-concentration *vs.* bond strength of dentin adhesives. *Dent Mater* 1992; 8: 229-233.
21. Cardoso P, Braga R, Carrilho M. Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. *Dent Mater* 1998; 14: 394-398.
22. Bowen RL. Adhesive bonding of various materials to hard tooth tissue. *J Am Dent Assoc* 1967; 44: 690-695.
23. Causton BE. Improved bonding of composite restorative to dentine. *Br Dent J* 1984; 156: 93-95.
24. Eliades GC, Caputo AA, Vougiouklakis GJ. Composition, wetting properties and bond strength with dentin of 6 new dentin adhesives. *Dent Mater* 1985; 1: 170-176.
25. Komatsu M, Finger W. Dentin bonding agents: Correlation of early bond strength with margin gaps. *Dent Mater* 1986; 2: 257-262.
26. Council on Dental Materials, Instruments and Equipment. Dentin bonding systems: An update. *J Am Dent Assoc* 1987; 114: 91-95.
27. Hinoura K, Moore BK, Phillips RW. Tensile bond strength between glass ionomer cements and composite resins. *J Am Dent Assoc* 1987; 114: 167-172.
28. Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG, Pashley DH. Nano-leakage: Leakage within the hybrid layer. *Oper Dent* 1995; 20: 18-25.
29. Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM, Pashley DH. Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesive. *J Dent Res* 1995; 10: 1679-1688.
30. Davidson CL, De Gee AJ, Feilzer A. The competition between the composite-dentin bond strength and the polymerization contraction stress. *J Dent Res* 1984; 63: 1396-1399.
31. Feilzer A, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. *J Dent Res* 1987; 66: 1636-1639.
32. Hegdahl T, Gjerdet NR. Contraction stresses of composite filling materials. *Acta Odontol Scand* 1977; 35: 191-195.
33. Bowen RL, Nemoto K, Rapson JE. Adhesive bonding of various materials to hard tooth tissue: Forces developing in composite materials during hardening. *J Am Dent Assoc* 1983; 106: 475-477.
34. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, Tay F. The microtensile bond test: A review. *J Adhes Dent* 1999; 1: 299-309.
35. Tam LE, Pilliar RM. Effects of dentin surface treatments on the fracture toughness and tensile bond strength of a dentin-composite adhesive interface. *J Dent Res* 1994; 73: 1530-1538.
36. Cardoso PE, Carrilho MR, Francci CE, Perdigao J. Microtensile bond strengths of one-bottle dentin adhesives. *Am J Dent* 2001; 14: 22-24.
37. Del Nero MO, De La Macorra JC. Sealing and dentin bond strengths of adhesive systems. *Oper Dent* 1999; 241: 94-202.
38. Frankenberger R, Perdigao J, Rosa BT, Lopes M. "No-bottle" *vs.* "multi-bottle" dentin adhesives. A microtensile bond strength and morphological studies. *Dent Mater* 2001; 17: 373-380.
39. Nunes MF, Swift EJ, Perdigão J. Effects of adhesive composition on microtensile bond strength to human dentin. *Am J Dent* 2001; 14: 340-343.
40. Tam LE, Pilliar RM. Fracture toughness of dentin/resin-composite adhesive interfaces. *J Dent Res* 1993; 72: 953-959.
41. Spohr AM, Ewerton N, Pacheco JFM. Tensile bond strength of four adhesive systems to dentin. *Am J Dent* 2001;14: 247-251.
42. Armstrong S, Boyer D, Keller J. Microtensile bond strength testing and failure analysis of two dentin adhesives *Dent Mater* 1998; 14: 44-50.
43. Yoshiyama M, Matsuo T, Ebisu S, Pashley D. Regional bond strengths of self-etching/self-priming adhesive systems. *J Dent* 1998;26:609-616.