Eur. J. Prosthodont. Rest. Dent., Vol.16, No. 4, pp 158-165
Printed in Great Britain

Methods Used in Dentine Bonding Tests: An Analysis of 102 Investigations on Bond Strength

FJ Trevor Burke*, Aftab Hussain†, Lawrence Nolan‡ and Garry JP Fleming§

Abstract - *Bond strength testing of resin-based materials to dentine may provide evidence as to the potential effectiveness of a dentine-bonding agent in producing adhesion and minimising microleakage. The purpose of this analysis was to analyse the methods used in 102 recently-published investigations on bond strength tests of resin composite to dentine. One hundred and two papers, published during the years 1998 to 2002 on the subject of bond strength measurement of resin composite to dentine were identified from a literature search. These papers were assessed for their inclusion of the following variables: type of test, thermal cycling regime, tooth notation, storage medium, preparation of dentine surface, rate of load application, type of dentine, film thickness, dimensions of surface area contact and time of testing. Shear testing was the most common type of test, used in 46% (n=47) of the papers analysed, human dentine was used in 77% (n=79) of the investigations, and 24 h post-placement was the predominant time of specimen testing (67%, n = 68). Surface area of contact, or specimen diameter, was stated in 65% (n=66) of the papers analysed. A number of variables were not stated in a majority of papers. There was little standardization of test methods and a number of potentially significant variables may not be reported or recorded in bond strength determinations which limit the overall standardization between different test centres.*

KEY WORDS: Bond strength testing, bond strength, microtensile testing

INTRODUCTION

Not only are the public in the developed world retaining their teeth for longer, but it would appear that they also increasingly request that their teeth are of good appearance[1,2]. As a result, it may be considered that the demand for aesthetic restorative treatment of anterior teeth is increasing. In most cases, aesthetic restorations are resin-based or formed in ceramic, and resin-based techniques have been considered appropriate for placement of such restorations[3]. A reliable means of bonding these aesthetic restorations to tooth substance is therefore paramount. Bonding to enamel has a long history of success, but satisfactory, reproducible bonding to dentine has proved more elusive, but is nevertheless central to the success of restorations which are bonded to tooth substance. While laboratory testing of materials is not a substitute for clinical evaluation, bond strength testing of resin-based materials to dentine may provide some evidence as to the potential effectiveness of a dentine-bonding agent in producing adhesion and minimising microleakage. The dental literature has highlighted many of the parameters that influence bond strengths of adhesive resins to dentine. Among these are:

- the degree of wetness/dryness of the substrate surface[4-7]
- the time of testing[8]
- age and depth of dentine[9-13]
- surface area of specimens[14]
- cutting instruments[15]
- storage medium and time of storage[16]
- operator[17].

Pashley and co-workers have additionally classified testing variables into broad groups, namely, substrate variables, etching variables, priming variables, storage variables and testing variables[18]. It would therefore appear to be essential that standardization of these variables occurs during bond strength testing.

Shear testing was identified, in 1997, to be most popular form of bond strength testing for resin-based materials to dentine[19], but has been criticised because the distribution of stresses along the interface is non-uniform, the stresses are sensitive to the loading geometry, the shape and size of the adherend and their relative elastic moduli[20]. Van Noort and co-workers, in 1989[20], pointed to the "pressing need for the development of a standard test for the measurement of bond strength of dentine bonding agents" with the potential benefit of comparability of tests from different laboratories. It appears that this cry went unheeded, since Stanley, in 1993[21], expressed anxieties about the variation in bond strength tests and the vagueness of detail in some work. This was, highlighted in 1997, with Al-Salehi and Burke[19] illustrating that there was little standardization of test methods, and that a significant number of variables were not recorded or not stated.

The purpose of this analysis was to summarise the methods used in recently-published investigations on bond strength tests of resin composite to dentine.

* DDS, MSC, MDS, FDS RCS(Edin.), FADM
† BDS
‡ BSc, BDS
§ BSc, PhD, FADM, ILTHE

METHODS AND MATERIALS

One hundred papers, published during the years 1998 to 2002, on the subject of bond strength measurement of resin composite to dentine were chosen from journals known to publish research on dentine bond strength testing, namely, Journal of Adhesive Dentistry, Dental Materials, American Journal of Dentistry, Operative Dentistry, Journal of Dentistry, and the Journal of Prosthetic Dentistry. These journals were available in the University of Birmingham Primary Dental Care Research Group and University of Birmingham School of Dentistry libraries. Additionally, a Medline search was carried out to identify papers on bond strength measurement of resin composite to dentine published in journals other than those above. The criteria for inclusion in this analysis were that the paper should include more than five of the following variables:

- Tensile, micro-tensile or shear test
- Thermal cycling
- Tooth notation
- Storage medium
- Preparation of dentinal surface
- Rate of load application/cross-head speed
- Type of dentine
- Film thickness
- Dimensions of surface area contact
- Time of testing from bonding material application
- Depth of dentine
- Condition of the dentine (moist or dry)
- Testing machine

Each paper was read by one of the authors (AH) in order to assess which of these potential variables had been recorded. In addition, other variables were noted if stated, namely, the type of adhesive resins tested, the modes of failure, sample size, number of operators and whether the specimens had been dried, and if so, for how long and at what distance. Where stated, the values for the bond strengths were also recorded. The results were then computerized for analysis.

RESULTS

A total of 102 papers (Table 1) were analysed.

The following results were established from this analysis.

Shear tests were the most common testing modality, used in 46% of the papers analysed. Microtensile testing was used in 31% of papers and tensile testing in 20%. The remaining papers used a mixture of tests.

Human dentine was used in 77% of the investigations analysed, while bovine dentine was used in 14%. Both types were used in two papers and monkey dentine in one. The type of dentine was not stated in 6 papers analysed.

The most commonly used teeth were molars (71%), while incisor teeth were used in 12% of papers, premolar teeth in 7% of papers, molars/incisors in two papers, and molars/premolars in one paper. The tooth type was not stated in 9 of the papers analysed.

Water/saline solutions were the most commonly used storage mediums, in 45 (44%) of the papers analysed. Chloramines were used in 22% of papers. A variety of media were used in other papers, including ethanol (n=2), formalin (n=7), sodium azide (n=9), thymol (n=1) and chlorhexidine (n=1). The storage medium was not stated in nine papers.

24 h was the predominant time of specimen testing (n = 68). Seven days was the second most prevalent time of testing, being used in 6 papers. A wide variety of other testing times was used, including 10 mins (n=1), 1 h (n=1), 12 h (n=1), 48 h (n=4), 72 h (n=1), 2 weeks (n=1) and 30-150 days (n=1). The time of testing was not stated in 12 of the papers analysed.

The surface area of contact, or specimen diameter, was stated in 66 papers (65%). In the remaining 36 papers (35%), the diameter of the specimen was not stated.

Silicon carbide abrasive paper was the commonly used method of dentine surface preparation, being used in 59% of the investigations. Other methods of preparation included diamond bur (n=12), sandpaper (n=7), and, aluminium oxide (n=6). The method of preparation of the dentine surface was not stated in 14 papers.

A variety of other test machines was used, including AG-500B (n=7) and Emic DL500 (n=6). The Instron was the most used testing machine (38%). The test machine was not stated in 36% of the papers analysed. The most prevalent loading rate, used in half the investigations was 1 mm/min. The next most frequently used rate of loading was 0.5 mm/min, which was used in just over a quarter papers. Among the other rates of loading utilised were 5 mm/min. (11%, and 2 mm/min(2%). The rate of loading was not stated in 6% of the papers analysed.

The sample size was stated in 89% of the papers, with the average sample size being 34 specimens. Sample size was not stated in 11 papers.

A number of potential variables were predominantly not stated, namely, film thickness, depth of dentine, moist or dry dentine/condition of the dentine, whether the specimen was dried (and if so, for how long), mode of failure, the number of operators involved in the tests, and whether or not thermal cycling was utilised.

Film thickness was not quoted in 87% of studies. Six stated that the resin layer "had been air thinned". A variety of other measures for film thickness were quoted, including "thin" (n=2), "various" (n=1), 38-41 µm (n=1), 40 µm (n=1), and 1 mm thickness (n=1).

The depth of dentine utilised in the studies was not stated in 54% the papers. Of the papers which stated the depth of dentine utilised, 17 stated that the dentine was "superficial", and thirteen that it was "mid-coronal" or "middle dentine". Smaller numbers of papers used "0.5 mm dentine removed" (n=6), 1.5 mm dentine depth (n=5), "coronal" (n=3) and "outer third of dentine removed" (n=3).

The degree of wetness/dryness of the test specimens was not stated in 53 papers (52%). Twenty-eight per cent of papers stated that the specimens had been left "moist" and seven "dry". Thirteen papers used both "dry" and "moist" specimens. Of the papers which provided details of the drying regimen, 28 stated the time for which drying took place and eight provided details of the distance of the air blast from the specimen. The most frequent time of drying, used in 16 papers, was 5 s, and the most frequently stated distance of drying (2 cm from the specimen surface) was used in two papers.

Table 1. *The 102 papers included in the study*

1. Kwong SM et al. Microtensile bond strengths to sclerotic dentine using a self etching and a total etching technique. Dental Materials, 2002; **18**: 359-369.

2. Franklin RT et al. Bonding of a self etching primer to non-carious cervicalsclerotic dentine : interfacial ultrastructure and microtensile bond strength evaluation. Journal of Adhesive Dentistry, 2000; **2**: 9-28.

3. Masatoshi et al. Bond strengths of self etching primer adhesives in in-vitro demineralised dentine following mineralising treatment. Journal of Adhesive Dentistry, 2000; **2**: 29-38.

4. Werner JF and Balkenhol M. Rewetting strategies for bonding to dry dentine with an acetone based adhesive. Journal of Adhesive Dentistry, 2000; **2**: 51-56.

5. Franklin RT et al. Mechanical disruption of dentine collagen fibrils during resin - dentine bond testing. Journal of Adhesive Dentistry, 2000; **2**:175-192.

6. Muench A et al. Influence of different dentinal substrates on the tensile bond strength of three adhesive systems. Journal of Adhesive Dentistry, 2000; **2**: 209-212.

7. Bedran de Costo AK et al. Influence of collagen removal on shear bond strength of one bottle adhesive systems in dentine. Journal of Adhesive Dentistry, 2000;**2**:271-277.

8.Asmussen E et al. The influence of relative humidity on the effect of dentine bonding systems. Journal of Adhesive Dentistry, 2001; **3**: 123-127.

9.Inoue H et al. Microtensile bond strength of two single step adhesive systems to bur-prepared dentine. Journal of Adhesive Dentistry, 2001; **3**: 129-136.

10. Nunes F et al. Effects of demineralisation depth on micro-tensile bond strength to human dentine. Journal of Adhesive Dentistry, 2001; **3**: 137-143.

11. Bell S et al. Bonding strength to two different surfaces of dentine under simulated pulpal pressure. Journal of Adhesive Dentistry, 2001; **3**: 145-152.

12. Inoue S et al. Microtensile bond strength of eleven contemporary adhesives to dentine. Journal of Adhesive Dentistry, 2001; **3**: 237-245.

13. Pereira GDS et al. How wet should dentine be? Comparison of methods to remove excess water during moist bonding. Journal of Adhesive Dentistry, 2001; **3**: 257-264.

14. Giannini M et al. The influence of tubule density and area of solid dentine on bond strength of two dentine adhesive systems to dentine. Journal of Adhesive Dentistry, 2001;**3**: 315-324.

15. Piemjai M et al. Effect of dentine conditioners on wet bonding of 4-META/MMA-TSB Resin. Journal of Adhesive Dentistry, 2001; **3**: 325-331.

16. Uno S et al. Microtensile bond strength evaluation of three adhesive systems in cervical dentine cavities. Journal of Adhesive Dentistry, 2001; **3**: 333-341.

17. Nikaido T et al. The in vitro bond strengths of two adhesive systems in class 1 cavities of human molars. Journal of Adhesive Dentistry, 2002; **4**: 31-39.

18. Fritz UB et al. Bonding efficiency of single bottle enamel/dentine adhesives. American Journal of Dentistry, 1999; **12**: 277-282.

19. Perdigao J. et al. Bond strengths of new simplified dentine-enamel adhesives. American Journal of Dentistry, 1999; **12**: 286-290.

20. Masatoshi et al. Bonding to caries affected dentine using self etching primers. American Journal of Dentistry, 1999; **12**: 309-314.

21. Braga RR et al. Tensile bond strength of filled and unfilled adhesives to dentine. American Journal of Dentistry, 2000; **13**: 73-76.

22. Miyazaki M et al. Effect of operator variability on dentin bond strength of two step bonding systems. American Journal of Dentistry, 2000; **13**: 101-104.

23. Cardoso PEC et al. Microtensile bond strengths of one bottle dentine adhesives. American Journal of Dentistry, 2001; **14**: 22-24.

24. Swift EJ Jr et al. Effects of restorative and adhesive curing methods on dentin bond strengths. American Journal of Dentistry, 2001; **14**: 137-140.

25. Dunn J et al. Comparison of shear and flexural bond strength tests versus failure modes of dentine bonding systems. American Journal of Dentistry, 2001; **14**: 297-303.

26. Pioch T et al. Effect of intrapulpal pressure simulation in vitro on shear bond strengths and hybrid layer formation. American Journal of Dentistry, 2001; **14**: 319-323.

27. Wahl AJ et al. Effect of water quality on the bonding of resin to moist dentine. American Journal of Dentistry, 2002; **15**: 114-116.

28. Saboia VP et al. Effect of collagen removal on shear bond strength of two single bottle adhesive systems. Operative Dentistry, 2000; **25**: 395-400.

29.Ogata M et al. Influence of the direction of tubules on bond strengths to dentine. Operative Dentistry, 2001; **26**: 27-35.

30.Gallo JR et al. Shear bond strength of four filled dentine bonding systems. Operative Dentistry, 2001; **26**: 44-47.

31.Gallo JR et al. Effect of delayed application on shear bond strength of four fifth-generation bonding systems. Operative Dentistry, 2001; **26**: 40-51.

32.Zheng L. et al. Relationship between adhesive thickness and microtensile bond strength. Operative Dentistry, 2001; **26**: 97-104.

33.Oberlander H. et al. Bond strength of polyacid modified resins using a new one step adhesive system. Operative Dentistry, 2001; **26**: 127-133.

34. Hannig M. et al. Composite to dentin bond strength, micromorphology of the bonded dentine interface and marginal adaptation of class 2 composite resin restorations using self etching primers. Operative Dentistry, 2001; **26**: 157-165.

35. Medina V et al. Effect of bonding variables on the shear bond strength and interfacial morphology of a one bottle adhesive. Operative Dentistry, 2001; **26**: 277-286.

36. Platt JA et al. The effect of double adhesive application on the shear bond strength to dentine of compomers using three one-bottle adhesive systems. Operative Dentistry, 2001; **26**: 313-317.

37. Ogata M et al. The effects of different burs on dentine bond strengths of self etching primer bonding systems. Operative Dentistry, 2001; **26**: 375-382.

38. Pilo R et al. Effect of preliminary treatment of the dentine surface on the shear bond strength of resin composite to dentine. Operative Dentistry, 2001; **26**: 569-575.

39. Seara SF et al. The influence of a dentin desensitiser on the microtensile bond strength of two bonding systems. Operative Dentistry, 2002; **27**: 154-160.

40. Kazemi RB et al. Effect of caries disclosing agents on bond strength of total-etch and self-etching primer dentine bonding systems to resin composite. Operative Dentistry, 2002; **27**: 238-242.

41. Miyazaki M et al. Influence of adhesive application duration on dentine bond strength of single application bonding systems. Operative Dentistry, 2002; **27**: 278-283.

42. Okuda M et al. Long term durability of resin dentin interface: nanoleakage versus microtensile bond strength. Operative Dentistry, 2002; **27**: 289-296.

43. Al-Ehaidels A et al. Shear bond strength of one bottle dentin adhesives. Journal of Prosthetic Dentistry, 2000; **84**: 408-412.

44. Hagge S. et al. Shear bond strength of an auto-polymerising core build-up composite bonded to dentine with 9 dentine adhesive systems. Journal of Prosthetic Dentistry, 2001; **86**: 620-623.

45. Prati C et al. Effect of removal of surface collagen fibrils on resin-dentine bonding. Dental Materials, 1999; **15**: 323-331.

46. Schneider BT et al. Dentine shear bond strength of compomers and composites. Dental Materials, 2000; **16**: 15-19.

47. Tanumiharja M. et al. Microtensile bond strengths of seven dentine adhesive systems. Dental Materials, 2000; **16**: 180-187.

48. Nakabayashi N. et al. Effect of etchant variation on wet and dry dentine bonding primed with 4-META/acetone. Dental Materials, 2000; **16**: 274-279.

49. Escribano N. et al. Sealing and dentine bond strength of adhesive systems in selected areas of perfused dentine. Dental Materials, 2001; **17**: 149-155.

50. Hara AT et al. Influence of cross-head speed on resin-dentine shear bond strength. Dental Materials, 2001; **17**: 165-169.

51. Armstrong SR et al. The influence of water storage and C-factor on the dentin-resin composite microtensile bond strength and debond pathway utilising a filled and unfilled adhesive resin. Dental Materials, 2001; **17**: 268-276.

52. Frankenberger R et al. No bottle versus multi bottle dentine adhesives – a micro-tensile bond strength and morphological study. Dental Materials, 2001; **17**: 373-380.

53. Burrow MF et al. A comparison of micro-tensile bond strengths of several dentine bonding systems to primary and permanent dentine. Dental Materials, 2002; **18**: 239-245.

54. Takahashi A et al. Effects of mechanical properties of adhesive resins on bond strength to dentine. Dental Materials, 2002; **18**: 263-268.

55. Nikaido T et al. Evaluation of thermal cycling and mechanical loading on bond strength of a self etching primer system to dentine. Dental Materials, 2002; **18**: 269-275.

Table 1. Continued over...

Table 1. Continued...

56. Pereira PNR et al. Effect of intrinsic wetness and regional difference on dentine bond strengths. Dental Materials, 1999; **15**: 46-53.

57. Perdigao J et al. The effect of a rewetting agent on dentine bonding. Dental Materials, 1999; **15**: 282-95.

58. Ruiz SG et al. Association between microtensile bond strength and leakage in the indirect resin composite/dentin adhesively bonded joint. Journal of Dentistry, 2001; **29**: 145-153.

59. Montes MA et al. A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats. Journal of Dentistry, 2001; **29**: 435-441.

60. Kitasako Y et al. Shear bond strengths of three resin cements to dentine over three years in vitro. Journal of Dentistry, 2001; **29**: 139-144.

61. Ruiz SG et al. Association between micro-tensile bond strength and leakage in the indirect resin composite/dentine adhesively bonded joint. Journal of Dentistry, 2001; **29**: 145-153.

62. Bouillaguet S. Bond strength of composite to dentine using conventional, one-step and self etch adhesive systems. Journal of Dentistry, 2001; **29**: 55-61.

63. Kitasako Y et al. Monkey pulpal response and micro-tensile bond strength beneath a one-application resin bonding system in vivo. Journal of Dentistry, 2000; **28**: 193-198.

64. Phrukkanon S et al. The effect of dentine location and tubule orientation on the bond strengths between resin and dentine. Journal of Dentistry, 1999; **27**: 265-274.

65. Yoshiyama M et al. Regional bond strengths of self-etching/self-priming adhesive systems. Journal of Dentistry, 1999; **27**: 609-616.

66. Spohr AM et al. Effect of refrigeration on tensile bond strength of three adhesive systems. Brazilian Dental Journal, 2001; **12**: 75-79.

67. Arbabzadeh F et al. Gap measurement and bond strength of five selected adhesive systems bonded to tooth structure. Australian Dental Journal, 1998; **43**: 175-180.

68. Cederlund A et al. Shear strength after ethylenediaminetetraacetic acid conditioning of dentin. Acta Odontologia Scandanavia, 2001; **59**: 418-422.

69. Nakajima M et al. Bond strengths of single-bottle dentin adhesives to caries-affected dentin. Operative Dentistry, 2000; **25**: 2-10.

70. Yoshikawa T et al. Effects of dentin depth and cavity configuration on bond strength. Journal of Dental Research, 1999; **78**: 898-905.

71. Soeno K et al. Effect of desensitisers on bond strength of adhesive luting agents to dentin. Journal of Oral Rehabilitation, 2001; **28**: 1122-1128.

72. Gernhardt CR. et al. Tensile bond strengths of four different dentin adhesives on irradiated and non-irradiated human dentin in vitro. Journal of Oral Rehabilitation, 2001; **28**: 814-820.

73.Iwami Y et al. Effect of enamel and dentin surface wetness on shear bond strength of composites. Journal of Prosthetic Dentistry, 1998; **80**: 20-26.

74. Paul SJ et al. Nanoleakage at the dentin adhesive interface vs micro-tensile nond strength. Operative Dentistry, 1999; **24**: 181-188.

75. Gallo JR et al. Shear bond strength to moist and dry dentin of four dentin bonding systems. American Journal of Dentistry, 2000; **13**: 267-270.

76. Gordan VV et al. Enamel and dentine shear bond strength of two resin modified glass ionomers and two resin based adhesives. Journal of Dentistry, 1998; **26**: 497-503.

77.Yoshiyama M et al. Regional bond strengths of self-etching/self-priming adhesive systems. Journal of Dentistry, 1998; **26**: 609-616.

78. Kanca J and Sandrik J. Bonding to dentin. Clues to the mechanism of adhesion. American Journal of Dentistry, 1998; **11**: 154-159.

79. Pioch T et al. Influence of different etching times on hybrid layer formation and tensile bond strength. American Journal of Dentistry, 1998; **11**: 202-206.

80. Jacobsen T et al. Effect of primer solvent, primer agitation, and dentin dryness on shear bond strength to dentin. American Journal of Dentistry, 1998; **11**: 225-228.

81. Leiskar J et al. In vitro shear bond strength of two resin composites to dentin with five different dentin adhesives. Quintessence International 1998; **29**: 787-792.

82. Issa MH et al. Strength of a 'no-bottle' adhesive system bonded to enamel and dentine. Dental Update 2000; **27**: 484-487.

83. Sperandio M et al. Effect of gamma radiation on dentin bond strength and morphology. Brazilian Dental Journal, 2001;**12**: 205-208.

84. Agostini FG et al. Bond strength of seld-etching primers to enamel and dentin of primary teeth. Pediatr. Dent 2001; **23**: 481-486.

85. Almuammar ME et al. Shear bond strength of six restorative materials. Journal of Clinical Pediatric Dentistry, 2001; **25**: 221-225.

86. Perdigao J et al. Effect of calcium removal on dentin bond strengths. Quintessence International 2001; **32**: 142-146.

87. Belli S et al. Regional bond strengths of adhesive resins to pulp chamber dentin. Journal of Endodontics, 2001; **27**: 527-531.

88. Moll K. et al. Effect of intrinsic and extrinsic moisture on bond strength to detine. Journal of Oral Rehabilitation, 2000; **27**: 149-164.

89. Price RBT et al. In vitro comparison of 10-minute versus 24-hour shear bond strengths of six dentin bonding systems. Quintessence International, 1999; **30**: 122-134.

90. Phrukkanon S et al. Effect of cross-sectional surface area on bond strengths between resin and dentin. Dental Materials, 1998; **14**: 120-128.

91. Schreiner RF. et al. Microtensile testing of dentin adhesives. Dental Materials 1998; **14**: 194-201.

92. Phrukannon S et al. The influence of cross-sectional shape and surface area on the microtensile bond test. Dental Materials, 1998; **14**: 212-221.

93. Cardoso PEC et al. Evaluation of micro-tensile, shear, and tensile tests determining the bond strength of three adhesive systems. Dental Materials, 1998; **14**: 394-398.

94. Barkmeier WW et al. Bond strength of composite to enamel and dentin using prime and bond 2.1. Operative Dentistry, 1999; **24**: 51-56.

95. Lucena-Martin C et al. Study of the shear bond strength of five one-component adhesives under simulated pulpal pressure. Operative Dentistry, 1999; **24**: 73-80.

96. Ogata M et al. Effect of dentin primer application on regional bond strength to cervical wedge-shaped cavity walls. Operative Dentistry, 1999; **24**: 81-88.

97. Benderli Y and Yucel T. The effect of surface treatment on the bond strength of resin composite to dentin. Operative Dentistry, 1999; **24**: 96-102.

98. Nero MOD et al. Sealing and dentin bond strengths of adhesive systems. Operative Dentistry, 1999; **24**: 194-202.

99. Manhart J et al. Bond strength of composite to dentin treated by air abrasion. Operative Dentistry, 1999; **24**: 223-232.

100. Hasegawa T et al. Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system. Operative Dentistry, 1999; **24**: 323-330.

101. Schilke R et al. Bovine dentin as a substitute for human dentin in shear bond strength measurements. American Journal of Dentistry, 1999; **12**: 93-95.

102. Hagge MS et al. Effect of refrigeration on shear bond strength of three dentin bonding systems. American Journal of Dentistry, 1999; **12**: 131-133.

The mode of failure was not stated in 54% of the papers analysed. Of the 47 papers which provided the mode of failure, this was adhesive in six, cohesive in four, and "both" in 37.

The number of operators used in production of the specimens was quoted in one paper, and not quoted in 101.

Whether thermal cycling was or was not used was stated in 18% of the papers analysed. 5-55º C for 625 cycles was the most frequently quoted regimen (n=5), with 5-55º C for 1000 cycles being used in four papers and a variety of other thermal cycling regimens being used in the other papers in which this information was provided.

A list of the types of resin material used and their frequency of use in all the investigations is presented in Table 2. Mean bond strength values are presented in Table 3.

Table 2. *Type of adhesive resin and numbers of papers in which these are utilised*

Name of material	*N*
Single bond	28
Scotchbond MP	23
Optibond solo	20
One step	19
Prime and bond 2.1	18
Prime and bond NT	16
Clearfil liner bond 2	12
Clearfil se bond	12
Clearfil liner bond 2V	10
Fluorobond	8
Gluma one bond	7
Syntac single component	7
Prompt-l-pop2	6
One coat bond	5
Etch and prime 3.0	5
Perma quick	4
EBS-Multi	3
One-up bond F	3
Unifil bond	3
A.R.T bond	2
All Bond 2	2
Bond 1	2
Macbond 2	2
A.R.T bond	2
All Bond 2	2
Solid bond	2
Liner bond 2	1
Liner bond 2V	1
Fuji bond LC	1
Universal se	1
Dentastic uno	1
Excite	1
Easybond	1
Monobond	1
Bistite 2 sc	1
Reactmer bond	1
Tenure quick	1
Tenure all surface	1
MZ-2000	1
Clearfil protect liner	1
Clearfil AP-X	1

Table 3. *Mean bond strength values reported for the 102 papers examined in the current investigation (values in brackets represent standard deviations).*

Testing Methodology	*Mean Bond Strength (MPa)*
Tensile	21.8 (19.1)
Microtensile	35.3 (17.2)
Shear	15.9 (5.8)

DISCUSSION

The production of a satisfactory bond to dentine has been, until recently, elusive. This is partly due to the biological characteristics of dentine, namely its high organic content, its tubular structure with the presence of odontoblastic processes, and the presence of the dentine smear layer[22]. This study presents an analysis of recent studies on bond strengths to dentine which have been published in peer-reviewed journals.

In the present study shear tests accounted for 46% of the investigations. In a similar study published in 1997[19], shear tests made up 80% of the investigations. This dramatic decrease could be due to the fact that many investigations that have been analysed show a large scatter in bond strength values for the same materials[20], thus researchers may have elected for other methods of testing. However, it is more likely that the emergence of a simplified non-labour intensive tensile testing methodology, namely, the micro-tensile testing method is the real reason. Interestingly, the micro-tensile testing method makes up 31% of the investigations analysed, and surpasses the more conventional tensile testing methodologies. The advantages of the micro-tensile method include the ease of simulation of the clinical situation (by building the restoration at the chair-side and then sectioning the specimen to get specimens of cross-sectional area approximately 1.6-1.8 mm^2 that produce a minimum scatter of results)[22] and the simplicity encourages the use of this testing methodology by non-experienced researchers.

Shear testing has been used to determine the bond strength of dental adhesives since Bowen described the method in 1965[18]. Problems arose, however, when the adhesive bonding became stronger and the test could no longer accurately measure the bond strength due to cohesive failure of the dentin before failure of the adhesive occurred. As the maximum limits of the shear testing method tend to be about 18-20 MPa, this led Sano to develop the micro-tensile testing method in 1994[24]. As the name implies, the microtensile testing method uses tensile forces rather than shear force to test the adhesives. According to Griffith's defect law the increasing size of a specimen will cause a reduction in the tensile strength seen, and this has been attributed to the greater number of defects in a larger sample. Tensile testing of adhesives has been studied but cohesive fractures of the dentin or enamel often take place, with values as low as 5 MPa being recorded by Perraka et al.[25]. This value is remarkable when studies by Bowen and Rodriguez showed the tensile strength of dentine to range between 52 and 104 MPa[26]. The studies by Sano revealed the failure of dentin at 71 MPa with the average between 50 and 60 MPa, which is a considerable improvement on 5 MPa and, as the failures were all adhesive, exhibited the tensile strength of dentin to be greater than 20MPa[24]. Microtensile testing uses an extremely small surface area of contact between the adhering surfaces, reducing the number of flaws and thereby increasing the values returned for the bond strength of the adhesives because the number of cohesive failures in the dentin is reduced. The results of the experiment by Sano showed that the tensile strength varied greatly depending upon the surface area used and suggested using 1.8-2 mm^2 due to the fact that it showed less scattering of results, at a value of below 2 mm^2 the number of cohesive failures of the dentin fell to zero[24]. The microtensile test method established by Sano in 1994 has been varied by different researchers. For example, Schreiner, in 1998,[27] used a cross head speed of 0.5 mm/min and Kwong[28] used a 0.45 mm^2 area of contact between the adhesive and the two opposing surfaces. A report by Kanemura[29] indicated that the original microtensile method devised by Sano was used on enamel, with the dimensions of the test specimens being the same as used for dentine testing.

The review of adhesion testing of dentin bonding agents by Pashley[18] and co-workers described the advantages and disadvantages of the technique, which are shown in Table 4. The results indicate that the disadvantages of the test method are far outweighed by the advantages, there is really no need to test bond strengths of below 5MPa for adhesives and if the samples are properly stored then dehydration should not be a problem. A paper by Phrukkanon et al[14] suggested the microtensile test gives results that represent the clinical situation much more accurately than the shear tests.

Table 4. *Advantages and disadvantages of the microtensile testing ethod (after Pashley et al.18)*

Disadvantages
1. Labour intensive, technically demanding
2. Difficult to measure bond strengths of below 5 MPa
3. Requires special equipment
4. Samples are so small that they dehydrate quickly
Advantages
1. More adhesive failures, fewer cohesive failures
2. Higher interfacial bond strengths can be measured
3. Permits measurements of regional bond strengths
4. Means and variances can be calculated for single teeth
5. Permits testing of bonds made to irregular surfaces
6. Permits testing of very small areas
7. Facilitates SEM examination of the failed bonds since the surface area is approximately 1 mm2

Tensile tests require large specimen sizes to facilitate gripping, and these sizes may not be representative of actual restorations. Shear tests test theoretical forces that can occur during mastication. However, they cannot replicate all oral conditions. The other factor to consider is that shear bond strengths vary considerably. Sudsaangiam and Van Noort have commented that the most popular methods of bond strengths do not correlate with clinical results and therefore should be interpreted with care.[30]

The publications which have been analysed in the present study have successfully been acquitted by the peer review process of the journals in which they were published. However, a number of significant variables were either not stated, or not actually recorded. The mode of failure was not stated in the majority (54%) of the investigations. Since mode of failure has been considered important[30] and has significance in the performance of a bonding agent and on the credibility of the bond strength data, it could be considered worrying that this information was not recorded routinely.

The type of dentine tested may be of considerable clinical significance. The commonest type of dentine utilised was superficial dentine, in 17% of the papers assessed. However, 54% failed to state the type of dentine. This is perplexing, since dentine is a substrate that varies in its properties at different depths through the tooth. Superficial dentine is a different substrate to deep dentine closer to the pulp, with Pashley et al in 1995 calculating that tubules make up 1% of the dentine close to the dentino-enamel junction but make up 22% of the dentine 1mm from the pulp[18]. Moreover, Konishi and co-workers have recently evaluated dentine shear strength, finding that the strength of dentine "near the pulp" was significantly lower than dentine in other locations[31]. Since it has previously been determined that bond strength values for the same material are different in different depths of dentine, it could be considered that the recording, and stating, of the type of dentine tested is an essential piece of information which must be included in a research paper on the subject.

Eighty-nine of the papers analysed (87%) failed to state the film thickness of the layer of bonding agent. Given that film thickness may affect bond strength values[32], it would appear that film thickness should be quoted, despite this being a different variable to standardise. Only three papers quoted the exact film thicknesses. Six papers quoted "air thinning" the adhesive, but previous research has demonstrated this to exert a deleterious effect on bond strength values[33].

Sixty-five per cent of the papers analysed stated the surface area of contact of the specimen to the dentinal surface. Conversely, 35% failed to include this information despite it being necessary to calculate the fracture strength of the specimens. In this respect, the fracture strength is calculated as the force required to break the bond divided by the surface area of the restorative material end and this is represented as megapascals (MPa). In this respect, it has previously been demonstrated that surface area of contact may affect bond strength, with highest bond strengths being obtained with the smallest area tested.[34]

Recent studies have commented on the state of the dentinal surface prior to the application of the adhesive. There is evidence that suggests some adhesives work better when the dentine surface is moist/damp while others work best when it is dry[5,6]. The state of the dentine surface in respect of wetness/dryness is therefore an important factor, which ideally should be stated. Yet 52% of the papers failed to state if the dentine was either tested dry, moist and/or both.

Another important factor, possibly of greatest relevance to the clinician, is the time of testing, given that bond strengths should be strong and immediate, in order to withstand the stresses encountered during polymerisation (and subsequent contraction) of resin-composite materials. For the clinician the most valuable bond strength measurements are at less than 5-10 mins post-placement, so bond strength tests should ideally be performed at a time as close as possible to this and further supplemented at 24 h, 1 month, 6 months and 1 year. However, only one investigation assessed in the present study carried out tests at 10 mins, finding that the bond strength was significantly less at 10 mins than at 24 h in three of the six materials tested[8]. However, in the present study, by far the most common time of testing was 24 h, with 74/110 (67%) of researchers favouring this time of testing. The clinical relevance of this time of testing must be questioned.

The method of preparation of the dentine surface has been shown to be of relevance to bond strength, yet 14%

of the papers assessed did not state how the dentine was prepared. There are reasons why specimen preparation is most readily accomplished by silicon carbide (SiC) paper (the most frequently used method in the papers assessed) rather than the burs that are commonly used in the mouth, but it would appear reasonable to suggest that this is another factor which should be recorded. Indeed, the SiC grit size should also be stated along with whether preparation occurred under water irrigation or not.

Thermal cycling presents an attempt at simulating intra-oral conditions, in which restorations are subjected to frequent changes in temperature. It may more frequently be associated with microleakage studies, but could be considered to present an additional challenge to specimens which are to be tested for bond strength. The results of the present study indicate that 82% of the papers did not provide information on whether the specimens had undergone thermal cycling. Of those that did state, the values ranged from 100 cycles to 2500 cycles with a mean range of 5-55° C. The majority of this 18% reported that they considered, from their own research, that thermal cycling had no significant effect on the final bond strength values. Different thermocycling regimes have been proposed to simulate the extreme conditions commonly experienced in the oral environment but at present there is little agreement amongst researchers regarding the temperature extremes that should be employed, the frequency of thermocycling, and the time that samples are exposed to each temperature extreme[35]. The maximum and minimum temperatures reliably recorded using thermocouples placed in the oral environment are 65 ± 1°C and 4 ± 1°C, respectively with closed mouth temperature of 37 ± 1°C[36,37], although these temperature extremes are expected to be encountered infrequently in service. In the oral environment high and low temperatures are only transient, and to simulate the clinical situation, a short dwell time (5s) should be used[38]. The frequency of the thermocycling regime should be based on the assumption that, at most, ten extreme thermocycles would occur per day. As a result, the 3500 cycles chosen would represent approximately one year of service for a restoration, which corresponds well with the experimental designs of previous investigators[35,38].

Rate of load application is another factor that showed great variability, varying from 0.5 to 5 mm/min. It is not possible to replicate the method by which force is applied in vivo: therefore it would appear reasonable to suggest the standardization of loading rate, given that none is actually appropriate to the clinical situation.

Lastly, operator variability may affect clinical performance of restorations and can influence laboratory test results[17]. It would therefore appear essential that the number of operators, and their status, is recorded.

The authors suggest that there should be more standardisation of test methods. It has previously been recommended[19] that shear/micro-tensile bond strength measurements of dentin bonding systems to resin composite be carried out:

1. On human molars/premolars
2. With saline storage medium at 37°C
3. With a diamond bur to prepare the dentinal surface
4. At a loading rate of 1 mm/min
5. After 5 mins, 24 h and at regular intervals up to 1 year
6. After thermocycling for 500 cycles of between 4-65° C
7. Under simulated physiological conditions of pulpal hydrostatic pressure

Ideally, the following variables should be recorded;

1. Number of thermal cycles
2. Film thickness and area of specimen contact to dentine
3. Dentinal depth
4. Condition of dentinal surface preparation and whether surface was wet or dry
5. Whether one or more operators carried out the work
6. Mode of failure
7. Bond strength values

Van Noort[20] and Pashley[18] and co-workers have respectively commented on the need for "standard test methods" and "new testing methods". Parameters related to the test design and experimental conditions should be easily controllable by the use of a standardised protocol, such as that recommended by ISO (TR 11405, 1994) for adhesion to dental tissues. Whilst it may be difficult to standardise all variables, researchers should, as a minimum set of data, provide the variables outlined in this article. If this suggestion is carried out, the relevance of the laboratory findings to the clinical situation may be enhanced and more true comparisons of test results may be possible. The results of the present study appear to have implications for laboratory testing of dental materials which are susceptible to clinically induced variability since a comparison between materials can only be achieved if specimen preparation occurs so that consistent reproducibility of results is produced for all materials under investigation and between test centres.

Sadly, there has been little improvement in standardisation of testing since a previous review of papers on dentine bond strength testing published in 1997[19]. It has recently been reported that bond strength numbers result from laboratory tests that have many inherent problems and an unknown relationship to clinical performance[39]. Further work is therefore needed to identify the parameters of *in vitro* testing that are most relevant to the clinical situation and apply these more regularly in testing methods. Dickens and Milos[40] concurred with this view in a recent paper which evaluated differences in strength and fracture surfaces following six shear bond test procedures using two primers. However, these workers concluded that, unless generally accepted standardisation was in place, the chisel-on-iris technique was the preferred method of testing as it avoided obvious extraneously introduced failure methods. The results of the present study indicate that this advice has not been widely accepted.

CONCLUSION

An analysis of 102 papers published on the subject of bond strengths of resin composite to dentine has indicated that there is little standardization of test methods and that a number of potentially significant variables may not be reported or recorded. These results have implications for laboratory testing of dental materials which are susceptible to clinically induced variability, since a comparison between materials can only be achieved if specimen preparation occurs wherein consistent reproducibility of results is produced for all materials under investigation and between test centres. Greater standardisation of test methods could improve the clinical relevance of studies on dentine-resin bond strength.

ADDRESS FOR CORRESPONDENCE

Prof. FJT Burke, E-mail:f.j.t.burke@bham.ac.uk.

REFERENCES

1. Reinhardt JW and Capilouto ML. Composite resin esthetic dentistry survey in New England. Journal of the American Dental Association, 1990; **120**: 541-544.
2. Burke F.J.T. Amalgam to tooth-coloured materials – implications for clinical practice and dental education: governmental restrictions and amalgam-usage survey results. J.Dent.2004:**32**:343-350.
3. Burke FJT, Fleming GJP, Nathanson D and Marquis PM. Are adhesive technologies needed to support ceramics? An assessment of the current evidence. Journal of Adhesive Dentistry, 2002; **4**: 7-22.
4. Elhabashy A and Swift EJ. Bonding to etched physiologically hydrated dentin. American Journal of Dentistry, 1994; **7**: 50-52.
5. Kanca J. Wet bonding:effect of drying time and distance. American Journal of Dentistry, 1996; **9**: 273-276.
6. Miears JR, Charlton DG and Hermesch CB. Effect of dentin moisture and storage time on resin bonding. American Journal of Dentistry, 1995; **8**: 80-82.
7. Nakajima M, Sano H, Zheng L, Tagami J and Pashley DH. Effect of moist vs dry bonding to normal vs caries-affected dentin with Scotchbond Multi-purpose Plus. Journal of Dental Research, 1999; **78**: 1298-1303.
8. Price RBT and Hall GC. In vitro comparison of 10-minute versus 24-hour shear bond strengths of six dentin systems. Quintessence International, 1999; **30**: 122-134.
9. Tao L and Pashley DH. Shear bond strengths to dentin:effects of surface treatments, depth and position. Dental Materials, 1988; **4**: 371-378.
10. Burrow MF, Takakura H, Nakajima M, Inai N, Tagami J and Takatsu T. The influence of age and depth of dentin on bonding. Dental Materials, 1994; **10**: 241-246.
11. McCabe JF and Rusby S. Dentine bonding agents – characteristic of bond strength as a function of dentine depth. Journal of Dentistry, 1992; **20**: 225-230.
12. Marshall GW, Marshall SJ, Kinney JH and Balooch M. The dentin substrate: structure and properties related to bonding. Journal of Dentistry, 1997; **25**: 441-458.
13. Yoshiyama M, Carvalho RM, Sano H, Horner JA, Brewer PD and Pashley DH. Regional bond strengths of resins to human root dentine. Journal of Dentistry, 1996; **24**: 435-442.
14. Phrukkanon S, Burrow MF and Tyas MJ. The influence of cross-sectional shape and surface area on the microtensile bond test. Dental Materials, 1998; 14: 212-221.
15. Sekimoto T, Derkson GD and Richardson AS. Effect of cutting instruments on permeability and morphology of the dentin surface. Operative Dentistry, 1999; **24**: 130-136.
16. Cooley RL and Dodge WW. Bond strength of three dentinal adhesives on recently extracted versus aged teeth. Quintessence International, 1989; **2**0: 513-516.
17. Sano H, Kanemura N, Burrow MF, Inai N, Yamada T and Tagami J. Effect of operator variability on dentin adhesion: students vs dentists. Dental Materials Journal, 1998; **17**: 51-58.
18. Pashley DH, Sano H, Ciucchi B, Yoshiyama M and Carvalho RM. Adhesion testing of dentin bonding agents: A review. Dental Materials, 1995; **11**: 117-125.
19. Al Salehi SK and Burke FJT. Methods used in dentin bonding tests: an analysis of 50 investigations on bond strength. Quintessence International, 1997; **28**: 717-723.
20. Van Noort R, Noroozi S, Howard IC and Cardew G. A critique of bond strength measurements. Journal of Dentistry, 1989; **17**: 61-67.
21. Stanley HR. An urgent plea for a Standardized Bonding (Adhesion) Test [guest editorial]. Journal of Dental Research, 1993; **72**: 1362-1363.
22. Lopes G, Baratieri LN, de Andrada MA and Vieiera LC. Dental adhesion: Present state of the art and future perspectives. Quintessence International, 2002; **33**: 213-224.
23. Burke FJT, Shortall AC, Coombe EC and Aitchison TC. Assessing restorative dental materials: 1. Test methods and assessment of results. Dental Update, 2002; **29**: 188-194.
24. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B,Carcalho R and Pashley DH. Relationship between surface area for adhesion and tensile bond strength – Evaluation of a micro-tensile bond test. Dental Materials, 1994; **10**: 236-240.
25. Perinka L, Sano H and Hosoda H. Dentin thickness, hardness and Ca-concentration vs. bond strength of dentin adhesion. Dental Materials, 1992; **8**: 229-233.
26. Bowen RL and Rodriguez MS. The long term durability of bond strengths of dentin. Journal of the American Dental Association, 1962; **64**: 378-387.
27. Schreiner RF, ChappellRP, Glaros AG and Eick JD. Microtensile testing of dentine adhesives. Dental Materials, 1998; **14**: 194-201
28. Kwong SM, Cheung GSP, Kei LH, Itthagarun A, Smales RJ, Tay FR and Pashley DH. Micro-tensile bond strengths to sclerotic dentin using a self-etching and total-etching technique. Dental Materials, 2002; **18**: 359-369
29. Kanemura N, Sano H and Tagami J. Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces. Journal of Dentistry, 1999; **27**: 523-530.
30. Sudsangiam S and Van Noort R. Do dentin bond strength tests serve a useful purpose? Journal of Adhesive Dentistry, 1999; **1**: 57-67.
31. Konishi N, Watanabe LG, Hilton JF, Marshall GW, Marshall SJ and Staninec M. Dentin shear strength: effect of distance from the pulp. Dental Materials, 2002; **18**: 516-520.
32. Unterbrinck G. Letter to the editor. Quintessence International, 1994; **25**: 521.
33. Causton BE and Sefton J. Some bonding characteristics of a HEMA/maleic acid adhesion promoter. British Dental Journal, 1989; **167**: 308-311.
34. Erickson RL, Glasspole EA and Retief DG. Influence of test parameters on dentin bond strength measurements Journal of Dental Research, 1989; **68**: 374.Absract No.1543.
35. Gale MS and Darvell BW. Thermal cycling procedures for laboratory testing of dental rstortions. Journal of Dentistry, 1999: **27**: 89-99.
36. Palmer DS, Barco MT and Billy EJ. Temperature extremes produced orally by hot and cold liquids. Journal of Prosthetic Dentistry, 1992; **67**: 325-7.
37. Spierings TM, Peters MB, Bosman F and Plasschaert AM. Verification of theoretical modelling of heat transmission in teeth by in vivo experiments. Journal of Dental Research, 1987; **66**: 1336-9.
38. Rossomando KJ and Wendt Jr SL. Thermocycling and dwell times in microleakage evaluation for bonded restorations. Dental Materials, 1995; **11**: 47-51.
39. Clinical Research Associates. Adhesives, do high shear numbers equal clinical success? CRA Newsletter, 2002; **26 (6)**: 1-2.
40. Dickens SH, Milos MF. Relationship of dentin shear bond strengths to different laboratory test designs. American Journal of Dentistry, 2002; **15**: 185-192.