

Dentin shear strength: effect of distance from the pulp

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Abstract

Objective: Dentin structure varies with orientation and location. Ultimate shear strength (USS) has also been found in previous studies to vary with location. The present study further explores this relationship between USS and various locations in coronal dentin as well as distance from the pulp.

Methods: Stick specimens were prepared from coronal dentin located in the center or under cusps of human molar teeth. These were tested in the shear mode at various distances from the pulp.

Results: Median values ranged from 52.7 (range 29.0–73.1) MPa near the pulp to 76.7 (range 53.9–104.0) MPa near the dentino–enamel junction. No differences were found among the buccal, central or lingual locations, however, the USS near the pulp was found to be significantly lower.

Significance: The properties of coronal dentin vary with distance from the pulp, which may affect adhesion and other aspects of restorative dentistry. © 2002 Academy of Dental Materials. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Dental materials; Dentin; Cohesion

1. Introduction

Dentin has a highly oriented tubule structure and the tubule number density and area fraction of intertubular dentin vary with distance from pulp. As is well-known, dentin is composed of about 50 vol% mineral, 30 vol% organic matter, and about 20 vol% fluid, and it consists of dentin tubules, peritubular dentin and intertubular dentin. The dentin tubule is surrounded by a highly mineralized peritubular dentin in a matrix of intertubular dentin consisting of collagen embedded with apatite crystals. The dentin tubules run radially from the pulp chamber to the dentino–enamel junction (DEJ) in the crown [1]. Using data from Garberoglio and Brannstrom [2], Pashley calculated the fractional areas occupied by tubules, peritubular matrix, and intertubular matrix at different distances from the pulp. He reported that tubule number density and peritubular dentin area decrease with distance from the pulp, and intertubular dentin area increases with distance from the pulp. He summarized this and other studies in a review

[3]. These components of dentin are well-known, but the values for dentin strength are variable because there are few studies that have sought to determine the relationship between the structure and the mechanical properties of dentin. Smith and Cooper used a punch method and reported values ranging from 39 MPa near the pulp to 131 MPa near the DEJ [4]. However, the shear testing method is far more popular and bending and friction complicated the method used by Smith and Cooper. Gwinnett reported the shear strength of dentin to be 36.2 MPa. He used relatively large samples from third molars, using a shear testing system, but he did not mention the location of tooth specimens [5]. Watanabe et al. reported the shear strength of dentin from the center of the crown, 78.0 MPa, was significantly weaker than that from the cuspal area, 91.8 MPa [6]. They demonstrated that the tubule orientation, and testing direction influenced dentin shear strength. Interestingly, although the fracture properties appear to be influenced by tubule orientation, the elastic properties do not [7]. The shear strength of dentin is important in understanding bonding to dentin, particularly when some dentin failures are reported. The properties of dentin are also of interest in understanding the clinical problems of tooth fractures and

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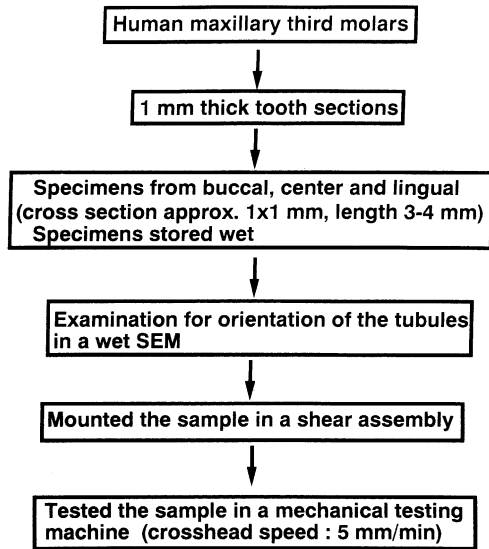


Fig. 1. Specimen preparation sequence.

in design consideration for biological or prosthetic replacement of dentin. The aim of the present study was to evaluate the influence of the distance from the pulp on shear strength of dentin at various locations. Understanding properties and characteristics of dentin more completely may lead to advances and greater success in restorative dentistry.

The hypothesis tested was that shear strength of dentin varies with location and distance from the pulp; the null hypothesis being that shear strength of dentin is the same at all locations and distances from the pulp. More specifically, we examined the hypothesis that there was no difference in dentin shear strength among buccal, central, and lingual locations of teeth, at three different depths: outer, middle, and pulpal.

2. Materials and methods

Twenty-two non-carious human maxillary third molars

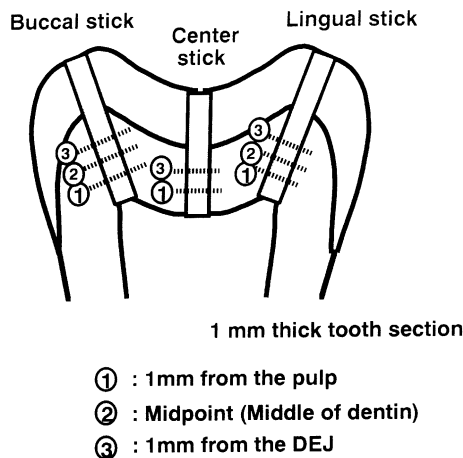


Fig. 2. Diagram of tested locations.

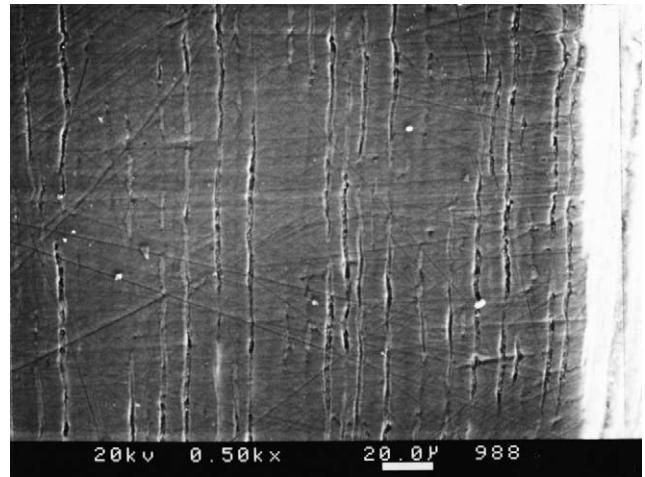
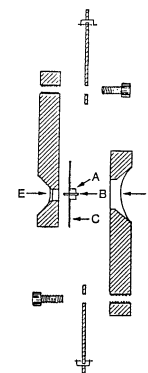


Fig. 3. SEM of a specimen, showing tubules oriented parallel to the side of the specimen and perpendicular to the fracture plane.

were used for this study. After extraction, these teeth were stored in de-ionized water for 2–4 years. Previous testing in our laboratory resulted in values similar to fresh teeth. The major steps of specimen preparation are diagrammed in Fig. 1. Sections 1 mm thick were cut from maxillary third molars through the center area midway between the mesio-buccal and disto-buccal cusp tips and through the center area midway between the mesio-lingual and disto-lingual cusp tips. We attempted to obtain three sticks from buccal, center and lingual locations. Final specimens (cross-section approximately 1 × 1 mm, length 3–4 mm) were taken from the desired locations, as shown in Fig. 2. Each specimen was briefly examined in a wet SEM (ISL SX40A with CFAS system that permits direct examination of wet uncoated specimens at approximately 100 mTorr) [8] to confirm orientation of the tubules in the desired direction, parallel to the long axis of the specimen. Specimens were



- A : Amalgam contained within a thin-walled tube
- B : Dentin specimen
- C : Mylar
- D : Dental stone
- E : Amalgam

Fig. 4. Single plane lap shear device.

Table 1
Dentin ultimate shear strength in MPa (*N* gives the number of USS measurements)

	Buccal	Central	Lingual	All
Outer				
Median	77.5	74.8	77.9	76.7
Range	(59.0–104.0)	(53.0–93.1)	(68.4–103.4)	(53.0–104.0)
<i>N</i>	11	11	11	33
Middle				
Median	72.3		81.1	73.9
Range	(61.4–95.0)		(52.8–95.7)	(52.8–95.7)
<i>N</i>	11	0	11	22
Pulpal				
Median	53.0	52.1	52.5	52.7
Range	(46.4–73.1)	(29.0–65.0)	(36.7–68.2)	(29.0–73.1)
<i>N</i>	11	11	11	33

rejected prior to testing if the tubules were not oriented in the desired direction. Fig. 3 shows an example of tubules oriented correctly, parallel to the side of the specimen and perpendicular to the fracture plane. Even the so-called wet SEM examination requires a partial vacuum, so some dehydration of specimens probably occurred. After SEM examination, the specimens were rehydrated overnight prior to testing. We assumed that this was effective, as only a brief period of re-hydration is effective in restoring the collapsed collagen matrix [9]. All testing was done on moist specimens, which were dried only briefly during mounting. All specimens were maintained in a wet environment until shear tested. Each specimen was mounted perpendicular to tubule direction in a single plane lap shear device (shown in Fig. 4) and coronal dentin shear strength was measured perpendicular to the long axis of the tubules. These specimens were tested at 1 mm from the pulp, 1 mm from the DEJ or at the midpoint, in a mechanical testing machine at a crosshead speed of 5 mm/min. At the central location, because of the limited thickness of dentin, only two depths were used: outer and pulpal.

3. Statistical methods

Eighty-eight measurements of USS were taken: 11 at each of three depths at the buccal and lingual locations, and 11 at each of two depths at the central location (1 mm from the DEJ and 1 mm from the pulp). At the central location all USS measurements came from separate teeth, whereas at the other locations measurements at two depths were taken in some teeth and at a single depth in others. These selections reflect specimen preparation difficulties, resulting from small specimen size and/or tubule orientation considerations. Of the 22 teeth evaluated at the central location, 17 were also evaluated at the lingual location and 18 at the buccal location. Because all measurements were not statistically independent, we used a mixed effects model to test the hypothesis: tooth was analyzed as a random effect

and depth and location as fixed effects. The depth was coded as an ordinal variable in anticipation of a decreasing trend in USS with greater depth (from the occlusal surface to the pulp); both linear and quadratic functions of depth were included in the model. The location was coded as a nominal variable since we expected no trend in USS by location. Finally, interactions between depth and location were included in the initial model to allow for differences among the locations in the depth trends; terms that were non-significant were dropped from the final model, which was used to estimate USS by depth and/or location.

4. Results

According to the mixed effects model including interaction terms, ultimate shear strength decreased nonlinearly with depth, as indicated by a highly statistically significant quadratic effect (Wald $P = 0.005$). There was no difference in USS, however, among the buccal, central and lingual locations, after controlling for depth (in intercepts, at the linear level, and at the quadratic level, Wald $P > 0.15$; see Table 1). Thus we excluded location terms from a subsequent model and estimated USS as a function of depth alone.

For all locations combined, the median (range) USS was 76.7 (53.0–104.0) MPa at the outer depth (1 mm from the DEJ), was 73.9 (52.8–95.7) MPa at the midpoint, and was 52.7 (29.0–73.1) MPa at the inner depth (1 mm from the pulp). These estimates, generated by a model that accounts for multiple USS measurements per tooth, are quite similar to simple estimates that do not account for the correlation in the data: 78.2 (11.6), 77.1 (11.5), and 53.0 (8.5) MPa, respectively. In fact, the variation between teeth represents only 14% of the total variation, indicating that the measurements within teeth are nearly independent.

Thus, the median dentin shear strength values show that strength is markedly lower at the pulpal depth than at the outer and middle depths, at all three tooth locations. At a given depth, the USS measurements are similar across all locations (Table 1).

5. Discussion

Morphological variations and the structural anisotropy of dentin might have an effect on its strength [1]. However, few studies have sought to determine the relationship between the structure and the mechanical properties of dentin. In this study, we evaluated the influence of the distance from the pulp on shear strength of dentin at various locations. The shear strength for the location 1 mm from the pulp was significantly lower than that for the location 1 mm from the DEJ and the midpoint. These results are in good agreement with most values reported in the literature. Smith and Cooper reported values varying from 39 MPa near the pulp

to 131 MPa near the DEJ [4]. These differences in shear strength of dentin result from the morphological characteristics of dentin [1]. They may also depend on the difference in the mechanical properties of intertubular dentin as shown using nanoindentation [10]. Gwinnett reported the shear strength of dentin to be 36.2 MPa and Pioch et al. obtained mean values of 38.6 MPa from human molars [5,11]. In both studies specimens were prepared by coring whole teeth and they found lower average values than the values in this study. We prepared small matchstick-like specimens, so that the difference between studies in specimen geometry and size might account for this lack of agreement. In bonding studies, the bonding area was found to influence the results, with smaller specimens giving higher values [12,13]. Watanabe et al. used the same shape specimens as the current study and reported values of 78.0 MPa near the midpoint at the central area of dentin and 91.8 MPa for the midpoint of the cusp area [6]. They speculated that Gwinnett had determined the shear strength of dentin closer to the pulp. They found a significant difference between the central dentin and the dentin under the cusps, in contrast to the present study. Watanabe et al. used freshly extracted teeth (<6 months old) for their study. The difference in the storage period might account for the lower values at the dentin under the cusps in shear strength (76.7), as compared with their study. Both the Gwinnett and Watanabe studies had values near 78 MPa for central dentin.

We found that dentin near the pulp was weaker in shear strength than at other locations, while similar differences have been reported in microhardness by Pashley et al. [14]. They correlated the microhardness of dentin with the tubule density. Recently, Kinney et al. determined nano-hardness of dentin as a function of intra-tooth location using a specially modified atomic-force microscope and reported that the hardness of peritubular dentin was independent of location, but the hardness of intertubular dentin did depend upon the location [10]. It is interesting that Kinney et al. demonstrated that most of the observed decrease in hardness can be accounted for by the decreased hardness of the intertubular dentin, and their results support the findings in this study.

The properties of dentin are important in understanding adhesion to dentin. Current dentin bonding systems exhibit shear strength values for adhesive/dentin bonds of around 20 MPa. It is supposed that the location and orientation of the dentin is important when bond strength testing is conducted [6]. Burrow et al. investigated the effects of dentin age and depth on tensile bond strength of three commercial bonding systems. They reported that specimens bonded to deeper dentin showed only slightly lower strengths, and concluded that dentin depth appeared to have little influence on the bond strength [15]. Burrow et al. speculated that the condition of the dentin specimens, which lacked pulpal fluid, might account for the reason why dentin depth did not influence tensile bond strength.

Furthermore, they suggested that the more recent bonding systems might be less influenced by dentin depth, and that the bond strength is more related to the quality of the resin-impregnated layer. Their study involved bonding systems that included acidic pre-treatments. Other types of materials, such as self-etching primers, may give different results. Further research is necessary to evaluate the relations between the location of the dentin and dentin bond strength. The effect of depth on bond strengths may also vary depending on the type of adhesive resin used [16]. Some studies have examined the structure of dentin as function of depth and have noted that because of a larger number and diameter of tubules near the pulp, less solid dentin is available for bonding [17,18]. The number and diameter of tubules may affect some bonding systems more than others; when the smear layer is left in place, the effect of the tubules may be smaller [19]. The effect of the tubules may also be related to pulpal pressure when the smear layer is removed [20].

When dentin bond strengths are evaluated, some dentin fractures are reported and they are often assumed to be due to improvements of dentin bonding systems, which have resulted in higher bond strengths. Such dentin failures have been interpreted to mean that possibly the ultimate strength of dentin is being approached [21]. However, the values of dentin bond strengths have been lower than the cohesive values of dentin [22]. Watanabe et al. demonstrated that such failures are likely to be the result of modifications of the dentin from the bonding procedure [6]. Another possibility is that in some tests the stress concentration is such that a crack is preferentially propagated into the dentin [23]. It must be noted that the modification of dentin from the bonding procedure is important when dentin bond strengths are evaluated.

More recently, microtensile testing for adhesion to dentin has become popular, because of the advantages it offers [24]. Sano et al. used microtensile testing to assess the relative contribution of mineral and collagen to the ultimate tensile strength of dentin [22]. Human coronal mineralized dentin gave a mean ultimate tensile strength (UTS) of 104 MPa. Bovine incisor coronal dentin exhibited UTS of 91 MPa, and bovine root dentin failed at 129 MPa. Those measurements are somewhat higher than the ones in the present study, but the method of testing is different as is the size of the specimen. The human dentin specimens were made from mid-coronal dentin and were tested in an orientation such that the fracture plane was parallel to the tubules.

Microtensile testing may be an appropriate additional test to evaluate location and orientation strength differences in dentin, because the smaller specimen size may make it possible to test regions that are difficult to test in the shear mode. It would also be desirable to relate the strength to other physical and chemical properties of dentin, such as degree of mineralization, microhardness, modulus of elasticity and others.

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References

- [1] Marshall Jr GW, Marshall SJ, Kinney JH, Balooch M. The dentin substrate: structure and properties related to bonding. *J Dent* 1997;25(6):441–58.
- [2] Garberoglio R, Brannstrom M. Scanning electron microscopic investigation of human dentinal tubules. *Arch Oral Biol* 1976;21(6):355–62.
- [3] Pashley DH. Dentin: a dynamic substrate—a review. *Scanning Microscopy* 1989;3:161–76.
- [4] Smith DC, Cooper WE. The determination of shear strength. A method using a micro-punch apparatus. *Br Dent J* 1971; 130(8): 333–7.
- [5] Gwinnett AJ. A new method to test the cohesive strength of dentin. *Quintessence Int* 1994;25(3):215–8.
- [6] Watanabe LG, Marshall GW, Marshall SJ. Dentin shear strength: effects of tubule orientation and intratooth location. *Dental Materials* 1996;12:109–15.
- [7] Kinney JH, Balooch M, Marshall GW, Marshall SJ. A micromechanics model of the elastic properties of human dentine. *Arch Oral Biol* 1999;44(10):813–22.
- [8] Marshall Jr GW, Staninec M, Torii Y, Marshall SJ. Comparison of backscattered scanning electron microscopy and microradiography of secondary caries. *Scanning Microsc* 1989;3(4):1043–9 (discussion 1049–50).
- [9] Marshall Jr GW, Wu-Magidi IC, Watanabe LG, Inai N, Balooch M, Kinney JH, et al. Effect of citric acid concentration on dentin demineralization, dehydration, and rehydration: atomic force microscopy study. *J Biomed Mater Res* 1998;42(4):500–7.
- [10] Kinney JH, Balooch M, Marshall SJ, Marshall Jr GW, Weihs TP. Atomic force microscope measurements of the hardness and elasticity of peritubular and intertubular human dentin. *J Biomech Eng* 1996;118(1):133–5.
- [11] Pioch T, Staehle HJ. Experimental investigation of the shear strengths of teeth in the region of the dentinoenamel junction. *Quintessence Int* 1996;27(10):711–4.
- [12] Phrukkanon S, Burrow MF, Tyas MJ. The influence of cross-sectional shape and surface area on the microtensile bond test. *Dent Mater* 1998;14(3):212–21.
- [13] Watanabe LG, Marshall GW, Marshall SJ. Variables influence on shear bond strengths testing to dentin. In: Granada International Symposium, Advanced Adhesive Dentistry, 1999 April 2000, Granada, Spain: Grafiche Erredue, Cirimido, Italy, 1999. p. 75–90.
- [14] Pashley D, Okabe A, Parham P. The relationship between dentin microhardness and tubule density. *Endod Dent Traumatol* 1985; 1(5):176–9.
- [15] Burrow MF, Takakura H, Nakajima M, Inai N, Tagami J, Takatsu T. The influence of age and depth of dentin on bonding. *Dent Mater* 1994;10(4):241–6.
- [16] Pashley EL, Tao L, Matthews WG, Pashley DH. Bond strengths to superficial, intermediate and deep dentin in vivo with four dentin bonding systems. *Dental Materials* 1993;9(1):19–22.
- [17] Olsson S, Oilo G, Adamczak E. The structure of dentin surfaces exposed for bond strength measurements. *Scand J Dent Res* 1993;101(3):180–4.
- [18] Suzuki T, Finger WJ. Dentin adhesives: site of dentin vs. bonding of composite resins. *Dent Mater* 1988;4(6):379–83.
- [19] Tam LE, Chan GP, Yim D. In vitro caries inhibition effects by conventional and resin-modified glass-ionomer restorations. *Oper Dent* 1997;22(1):4–14.
- [20] Tao L, Tagami J, Pashley DH. Pulpal pressure and bond strengths of SuperBond and Gluma. *American Journal of Dentistry* 1991;4(2):73–76.
- [21] Kanca J. Dental adhesion and the All-Bond system. *Journal of Esthetic Dentistry* 1991;3(4):129–32.
- [22] Sano H, Ciucchi B, Matthews WG, Pashley DH. Tensile properties of mineralized and demineralized human and bovine dentin. *Journal of Dental Research* 1994;73(6):1205–11.
- [23] Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. *J Dent* 1989;17(2):61–7.
- [24] Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, et al. The microtensile bond test: a review. *J Adhes Dent* 1999;1(4):229–309 [In Process Citation].