

# The Effect of a Resin-based Sealer Cement on Micropunch Shear Strength of Dentin

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## Abstract

Dentinal tubules occupy a substantial proportion of total dentin volume, especially of inner dentin. Resin-based sealer cements are known to penetrate deeply into dentinal tubules, but their ability to influence root strength is controversial. In this study, the contribution of dentinal tubules to shear strength and the influence of a resin-based sealer on shear strength were evaluated. Coronal 100- $\mu\text{m}$  sections of 12 single-canal premolar teeth were tested in different locations (buccal and proximal) and tubule directions (parallel and perpendicular) using the micropunch shear test (MPSS). Tests were also conducted by using 10 two-canal premolars, with one untreated canal and the other obturated using epoxy resin-based sealer (plus gutta-percha). No difference in MPSS was found because of location or tubule orientation ( $p > 0.05$ ). Outer dentin had a higher MPSS than middle and inner dentin ( $p < 0.001$ ). Tubule infiltration by epoxy resin did not increase MPSS. (*J Endod* 2008;34:1215–1218)

## Key Words

Dentinal tubules, punch shear strength, resin infiltration, sealer cement

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Dentinal tubules are the most characteristic structural feature of dentin. The number of dentinal tubules varies in different locations, from approximately 20,000/mm<sup>2</sup> near the dentinoenamel junction (DEJ), increasing to over 45,000/mm<sup>2</sup> near the pulp in coronal dentin. The diameter of each dentinal tubule is approximately 0.9  $\mu\text{m}$  at the DEJ, increasing to about 2.5  $\mu\text{m}$  at the pulpal surface (1). The percentage of dentin occupied by dentinal tubules varies from less than 1% in the outer dentin to more than 22% near the pulp (2). Despite their distinctive appearance, the effect of tubule density, diameter, and orientation on physical properties of dentin remains controversial. Both ultimate tensile strength and work of fracture are dependent on tubule orientation (3–5), with higher values for fracture parallel to tubule direction. Punch shear strength is greater in outer versus inner dentin (6, 7), but the effect of tubule orientation has not been previously reported. Root dentin has a higher tensile strength than coronal dentin (8, 9), and the difference has been attributed to the lower number of dentinal tubules in root dentin.

Several recent studies have documented the extensive penetration of resin-based sealers into dentinal tubules in root canal-treated teeth (10–13). The resin penetrates not only the main tubules but also tubule branches (11). Several studies have also claimed that the use of a resin-based root-filling material (Resilon; Pentron, Wallingford, CT) reinforced roots against fracture (14–16), although other studies have not confirmed this finding (17–19). Bond strength of resin-based sealers to dentin has also been variable (20–22). Extensive resin infiltration of tubules has the potential to increase fracture strength of dentin. Thus, the aim of this study was to investigate the role of tubules in biomechanical properties of dentin, as measured by micropunch shear strength (MPSS), and to evaluate the effect of resin infiltration on strength. The null hypotheses tested were as follows: (1) there is no difference in MPSS of coronal root dentin in relation to location (buccal and proximal), tubule orientation (perpendicular and parallel), and area (inner, middle, and outer dentin), and (2) there is no difference in MPSS of resin-filled root dentin and sound dentin.

## Materials and Methods

This study was conducted using maxillary and mandibular premolars with either a single root (12 teeth) or two roots (10 teeth), extracted for orthodontic reasons. Teeth were stored in 1% chloramine T (Sigma-Aldrich, St Louis, MO) at 4°C until use. The protocol was approved by the Human Research Ethics Committee, University of Melbourne, Australia. Teeth were thoroughly cleaned, and the crown of each tooth was resected 2 mm apical to the cemento-enamel junction. All samples were then prepared from the coronal third of the root.

## Sample Preparation

### Transverse Sections

A sample cut in transverse section approximately 200  $\mu\text{m}$  thick was lapped and polished with a series of silicon carbide papers (1,200, 2,400, and 4,000 grit) until the thickness was 100  $\mu\text{m}$  as measured with a digital caliper to the nearest 0.001 mm. Care was taken to prevent desiccation of the dentin, and tooth slices were stored moist at 4°C until the punch shear test. These samples allowed testing perpendicular to tubule orientation.

**Longitudinal Samples**

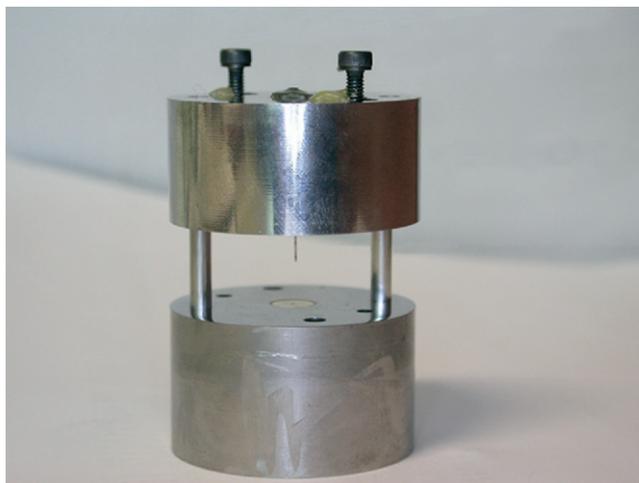
The remaining portion of the coronal third of the root was used for longitudinal sections, which were tested in a direction parallel to tubule orientation. The sample was attached to a mounting stub and sliced into buccal, lingual, and proximal samples. Each sample was then sequentially cut to yield three samples: inner, middle, and outer dentin. All samples were lapped to a thickness of 100  $\mu\text{m}$  with silicon carbide papers as described earlier. Before testing, all sections were examined under a light microscope to locate regions in which the dentinal tubules were seen precisely in a cross or longitudinal section.

**Punch Shear Testing**

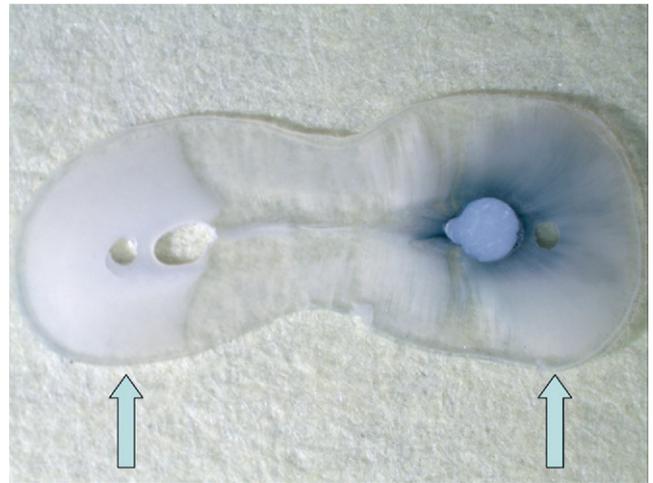
A micropunch shear apparatus was modified from the device previously described (23) with a punch diameter of 0.3 mm (Fig. 1). The dentin slice was positioned over the central hole of the lower die, and the upper die was placed over the lower die and secured so that the dentin slice was constrained. All tests were randomized in sequence among inner dentin (approximately 0.5 mm from the canal space), middle dentin, and outer dentin (approximately 0.5 mm from the root surface). The punch apparatus was then mounted on a universal testing machine (Instron model 5544; Instron Corp, Canton, MA). The load was applied to the punch at a constant crosshead speed of 0.1 mm/min until a hole was punched, as determined from the drop in force. The MPSS was calculated by the following equation: shear strength (MPa) = force/ $\pi \times$  diameter  $\times$  thickness. Force is in newtons, and punch diameter and specimen thickness are in millimetres. Data were normally distributed and were analyzed by general linear model analysis of variance with location (buccal and proximal), orientation of tubules (parallel and perpendicular), and area tested (inner, middle, and outer dentin) as independent variables. Post hoc pair-wise comparisons were conducted by using Tukey multiple comparisons. For each outcome, statistical significance was set at  $p < 0.05$ .

**Resin-infiltrated versus Sound Dentin**

This part of the study investigated the effect on MPSS in locations in which sealer penetration was confirmed by incorporating Sudan black into an epoxy resin-based sealer cement (AH Plus; Dentsply DeTrey, Konstanz, Germany) (13). Ten premolars with two separate canals were used. For each tooth, buccal and palatal canals were randomly allocated



**Figure 1.** The micro-punch apparatus with 300  $\mu\text{m}$  diameter punch. After the dentin slice was positioned over the bush of the lower die, the upper die was placed over the lower die and secured with screws so that the dentin slice was constrained.



**Figure 2.** The right hand side shows the canal filled with gutta percha and AH Plus™ stained with Sudan black. The sealer extensively infiltrated the inner third of dentin where the punch shear strength was measured. A comparable area was tested in the canal with sound dentin on the left hand side. The mean MPSS of dentin filled with AH Plus™ was slightly higher than sound dentin, with no statistically significant difference ( $p = 0.08$ ).

(using a random numbers table) to either no treatment (sound dentin) or prepared canal with a 0.04 taper ProFile (ProFile; Dentsply Maillefer, Johnson City, TN) to master apical rotary size #40, plus root filling with main cone plus AH Plus with Sudan black. Sections from the coronal third of each root were lapped in series with silicon carbide paper until the thickness was approximately 100  $\mu\text{m}$ , as described earlier. An area of inner dentin in which sealer containing Sudan black was seen heavily infiltrating tubules was tested for MPSS, and a comparable area was tested in the canal with sound dentin (Fig. 2). The effect of epoxy resin sealer on MPSS was analyzed by using a paired *t* test.

**Results**

The mean MPSS of coronal root dentin in two locations (buccal and proximal), in two orientations (parallel and perpendicular), and in three areas tested (inner, middle, and outer) are shown in Table 1. The mean value of MPSS varied from  $73.6 \pm 18.3$  to  $117.5 \pm 18.8$  MPa. There were no statistically significant differences in MPSS between the two locations (buccal and proximal) ( $p = 0.28$ ) or when comparing the two orientations with respect to dentinal tubules ( $p = 0.6$ ). However, the mean MPSS of outer dentin was significantly higher than that of the middle and inner dentin ( $p < 0.001$ ), and the middle dentin had a significantly higher MPSS than the inner dentin ( $p < 0.01$ ) (Table 1).

The test was conducted for roots filled using epoxy resin-based sealer, with Sudan black as an indicator of areas in which tubules were densely filled with resin (Fig. 2). No difference in MPSS of inner dentin in which color could be seen was found compared with sound dentin from the same tooth in a comparable location (root-filled =  $74.5 \pm 10.8$  MPa, sound dentin =  $67.8 \pm 11.5$  MPa,  $p = 0.08$ ).

**Discussion**

Using the punch shear test to show dentin strength was first reported by Roydhouse in 1970 (24). The shear strength of dentin varied from 68.6 MPa to 147 MPa depending on the site within the tooth, tooth type, and the direction of the punch in relation to the long axis of the tooth. Smith and Cooper (7) used a micropunch 100, 200, or 300  $\mu\text{m}$  in diameter and reported that the value near the DEJ was higher than near the pulp. Dentin from root-filled teeth has been variously reported

**TABLE 1.** Mean Values of MPSS in Different Locations, Areas, and Orientations in Relation to Dentinal Tubules (Mean  $\pm$  SD, n = 12)

Mean MPSS (MPa) (Mean $\pm$ SD)	Area Tested	Inner Dentin	Middle Dentin	Outer Dentin
Perpendicular to dentinal tubules	Buccal	83.1 $\pm$ 8.1	90.0 $\pm$ 7.7	102.1 $\pm$ 14.5
	Proximal	95.8 $\pm$ 11.2	96.6 $\pm$ 19.7	100.0 $\pm$ 13.9
Parallel to dentinal tubules	Buccal	73.6 $\pm$ 18.3	96.4 $\pm$ 17.7	117.5 $\pm$ 18.8
	Proximal	77.7 $\pm$ 21.3	95.9 $\pm$ 20.1	115.48 $\pm$ 25.0

MPSS, micropunch shear strength.

The mean MPSS of outer dentin was highly significantly different from that of inner and middle dentin ( $p < 0.001$ ). There was also a statistically significant difference between inner and middle dentin ( $p < 0.01$ ).

to be slightly weaker than vital dentin (25) or not different (23). The present study modified the punch apparatus from Sedgley and Messer (23) to a smaller diameter punch (0.3 mm), and mean MPSS values were similar to those reported previously.

Among the three factors investigated (area, location, and tubule orientation), only one factor led to a statistically significant difference in the MPSS, namely area (inner, middle, and outer). MPSS showed a statistically significant decrease from outer to middle and inner dentin. This confirms previous work (6, 7) and parallels many studies reporting higher microhardness of outer compared with inner dentin (26–28). This might be explained by the lower volume occupied by tubules in outer dentin and the corresponding increase in the amount of intertubular dentin (as well as the greater quantity of peritubular dentin in outer dentin). In addition, lower hardness and stiffness values of the intertubular dentin near the pulp have also been found (29). No difference in MPSS was found in relation to the two orientations of applied force in relation to dentinal tubule direction. Watanabe et al. (30) also reported that the shear strength (not punch shear) of midcoronal dentin was unaffected by tubule direction. This result is in contrast to tensile strength (3) and work of fracture (4, 5), which were greater when the fracture was in a direction parallel to tubules than when it was perpendicular to tubules.

The potential effect of resin infiltration on MPSS of root dentin was also investigated. Sudan black was incorporated into AH Plus as an indicator of areas in which tubules were densely filled with resin (13). No difference in MPSS of inner dentin in which color could be seen was found compared with sound dentin from the same tooth in a comparable location. The physical properties of the resin may be too low in comparison with those of dentin to produce a significant strengthening effect. In addition, the filler particles present in sealer cements are generally too large to enter the tubules (21) so that the material filling the tubules is largely unfilled resin, with correspondingly low strength. Whether an alternative resin-based sealer (Resilon) would have resulted in a strengthening effect, such as has been reported for resistance to root fracture (14–16), is unknown; the more variable tubule penetration by Resilon relative to AH Plus (21) is likely to result in a less predictable effect on strength.

The correlation of sealer penetration into the dentinal tubules with interfacial bond strength is also of interest. Tao and Pashley (31) suggested that increased bond strength is not correlated with dentinal tubule tag formation (in coronal dentin). They calculated that 85% of the bond strength resulted from the hybrid layer and only 15% from resin tags. With root canal fillings, however, the volume of inner dentin occupied by tubules is much greater than in coronal outer dentin. Thus, the contribution of resin tags to bond strength should be much greater than in the case of coronal restorations. Mannocci et al. (32) reported that no statistically significant difference was found between apical and coronal sections regarding the presence of hybrid layer or the penetration of resin into the dentinal tubules. This confirms the suggestion that micromechanical retention by penetration of sealer tags inside the tubules may not be an important factor affecting adhesion of root canal sealers to

dentin (33). Thus, a major role for sealer cements in strengthening the root against fracture must be questioned.

## Conclusions

Using MPSS, no difference in punch shear strength of dentin was observed because of location (buccal or proximal) or to tubule orientation (perpendicular or parallel). Only the area tested (inner, middle, or outer) was found to show a difference. Epoxy resin infiltration of tubules did not increase shear strength of dentin.

## References

- Garberoglio R, Brännström M. Scanning electron microscopic investigation of human dentinal tubules. *Arch Oral Biol* 1976;21:355–62.
- Pashley DH. Clinical correlations of dentin structure and function. *J Prosthet Dent* 1991;66:777–81.
- Lertchirakarn V, Palamara JE, Messer HH. Anisotropy of tensile strength of root dentin. *J Dent Res* 2001;80:453–6.
- Rasmussen ST, Patchin RE. Fracture properties of human enamel and dentin in an aqueous environment. *J Dent Res* 1984;63:1362–8.
- Rasmussen ST, Patchin RE, Scott DB, Heuer AH. Fracture properties of human enamel and dentin. *J Dent Res* 1976;55:154–64.
- Chng HK, Palamara JE, Messer HH. Effect of hydrogen peroxide and sodium perborate on biomechanical properties of human dentin. *J Endod* 2002;28:62–7.
- Smith DC, Cooper WE. The determination of shear strength. A method using a micropunch apparatus. *Br Dent J* 1971;130:333–7.
- Mannocci F, Pilecki P, Bertelli E, Watson TF. Density of dentinal tubules affects the tensile strength of root dentin. *Dent Mater* 2004;20:293–6.
- Inoue T, Takahashi H, Nishimura F. Anisotropy of tensile strengths of bovine dentin regarding dentinal tubule orientation and location. *Dent Mater J* 2002;21:32–43.
- Bergmans L, Moisiadis P, De Munck J, Van Meerbeek B, Lambrechts P. Effect of polymerization shrinkage on the sealing capacity of resin fillers for endodontic use. *J Adhes Dent* 2005;7:321–9.
- Mamootil K, Messer HH. Penetration of dentinal tubules by endodontic sealer cements in extracted teeth and in vivo. *Int Endod J* 2007;40:873–81.
- Patel DV, Sherriff M, Ford TRP, Watson TF, Mannocci F. The penetration of RealSeal primer and Tubliseal into root canal dentinal tubules: a confocal microscopic study. *Int Endod J* 2007;40:67–71.
- Weis MV, Parashos P, Messer HH. Effect of obturation technique on sealer cement thickness and dentinal tubule penetration. *Int Endod J* 2004;37:653–63.
- Teixeira FB, Teixeira EC, Thompson JY, Trope M. Fracture resistance of roots endodontically treated with a new resin filling material. *J Am Dent Assoc* 2004;135:646–52.
- Schäfer E, Zandbiglari T, Schäfer J. Influence of resin-based adhesive root canal fillings on the resistance to fracture of endodontically treated roots: an in vitro preliminary study. *Oral Surg Oral Med Oral Pathol Radiol Endod* 2007;103:274–9.
- Hammad M, Qualtrough A, Silikas N. Effect of new obturating materials on vertical root fracture resistance of endodontically treated teeth. *J Endod* 2007;33:732–6.
- Carvalho CA, Valera MC, Oliveira LD, Camargo CH. Structural resistance in immature teeth filling root reinforcements in vitro. *Dent Traumatol* 2005;21:155–9.
- Stuart CH, Schwartz SA, Beeson TJ. Reinforcement of immature roots with a new resin filling material. *J Endod* 2006;32:350–3.
- Wilkinson KL, Beeson TJ, Kirkpatrick TC. Fracture resistance of simulated immature teeth filled with resilon, gutta-percha, or composite. *J Endod* 2007;33:480–3.
- Fisher MA, Berzins DW, Bahcall JK. An in vitro comparison of bond strength of various obturation materials to root canal dentin using a push-out test design. *J Endod* 2007;33:856–8.
- Jainena A, Palamara JEA, Messer HH. Push-out bond strengths of the dentine-sealer interface with and without a main cone. *Int Endod J* 2007;40:882–90.
- Sly MM, Moore BK, Platt JA, Brown CE. Push-out bond strength of a new endodontic obturation system (resilon/epiphany). *J Endod* 2007;33:160–2.

23. Sedgley CM, Messer HH. Are endodontically treated teeth more brittle? *J Endod* 1992;18:332–5.
24. Roydhouse RH. Punch-shear test for dental purposes. *J Dent Res* 1970;49:131–6.
25. Carter JM, Sorensen SE, Johnson RR, Teitelbaum RL, Levine MS. Punch shear testing of extracted vital and endodontically treated teeth. *J Biomech* 1983;16:841–8.
26. Fuentes V, Toledano M, Osorio R, Carvalho RM. Microhardness of superficial and deep sound human dentin. *J Biomed Mater Res A* 2003;66:850–3.
27. Saleh AA, Ettman WM. Effect of endodontic irrigation solutions on microhardness of root canal dentine. *J Dent* 1999;27:43–6.
28. Toledano M, Osorio R, Osorio E, Prati C, Carvalho RM. Microhardness of acid-treated and resin infiltrated human dentine. *J Dent* 2005;33:349–54.
29. Kinney JH, Balooch M, Marshall SJ, Marshall JGW, Weihs TP. Hardness and young's modulus of human peritubular and intertubular dentine. *Arch Oral Biol* 1996;41:9–13.
30. Watanabe LG, Marshall GW Jr, Marshall SJ. Dentin shear strength: effects of tubule orientation and intratooth location. *Dent Mater* 1996;12:109–15.
31. Tao L, Pashley DH. Shear bond strengths to dentin: effects of surface treatments, depth and position. *Dent Mater* 1988;4:371–8.
32. Mannocci F, Innocenti M, Ferrari M. Stereomicroscopic and scanning electron microscopic study of roots obturated with vertically condensed gutta-percha, epoxy resin cement, and dentin bonding agent. *J Endod* 1998;24:397–400.
33. Saleh IM, Ruyter IE, Haapasalo M, Orstavik D. The effects of dentine pretreatment on the adhesion of root-canal sealers. *Int Endod J* 2002;35:859–66.