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# Ultimate tensile strength of tooth structures

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## KEYWORDS

Tensile strength;  
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enamel junction

**Summary Objective.** This study determined the ultimate tensile strength (UTS) of enamel (E), dentin (D) and dentin-enamel junction (DEJ) using the microtensile technique. It was hypothesized that the UTS of dental structures varies according to location and nature.

**Methods.** Intact occlusal enamel surfaces from extracted human third molars were etched with 37% phosphoric acid and bonded with a one-bottle adhesive system. The bonded occlusal surfaces received a resin composite build-up and teeth were serially, vertically sectioned into several 0.7 mm thick slabs. Each slab was then trimmed to a dumbbell-shaped specimen with irrigated diamond burs to reduce the cross-sectional area to approximately 0.5 mm<sup>2</sup> at E, D or DEJ. E was tested according to its prismatic orientation (parallel, EP; and transversally, ET) and D as function of depth (superficial, DS; middle, DM and deep, DD). Specimens were tested in tension in an Instron testing machine at 0.5 mm/min. Results were analyzed by one-way ANOVA and Duncan's Multiple Range test.

**Results.** UTS mean values ( $N = 20$ ) were, MPa (SD): DEJ, 46.9 (13.7)<sup>b</sup>; EP, 42.1 (11.9)<sup>b</sup>; ET, 11.5 (4.7)<sup>d</sup>; DS, 61.6 (16.2)<sup>a</sup>; DM, 48.7 (16.6)<sup>b</sup> and DD, 33.9 (7.9)<sup>c</sup>. Enamel stressed transversally to its prismatic orientation was significantly weaker ( $p < 0.05$ ). Dentin depth significantly affected its UTS ( $p < 0.05$ ). DEJ presented UTS that was similar to EP and DM ( $p > 0.05$ ).

**Significance.** The UTS of dental structures varies according to its nature and location.

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## Introduction

The tooth is the only mineralized organ that is located partially internal and partially external to the body. To minimize wear during function,

the tooth includes highly mineralized tissues that present physical properties based on their composition and micro morphology.<sup>1</sup> The anatomical crowns of teeth are covered by dental enamel, which is the hardest tissue in the body, and is composed of 92-96% of inorganic matter, 1-2% of organic material and 3-4% of water by weight.<sup>1</sup> Most of the inorganic matter is hydroxyapatite that is contained in the basic structural unit of enamel,

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the rod or prism.<sup>2</sup> Enamel hardness is attributed to its high mineral content<sup>3</sup> and the brittle property is due to its high elastic modulus and low tensile strength.<sup>4</sup> Clinically, enamel cracks and fracture can occur despite enamel being a very strong substrate. Studies have shown that enamel is anisotropic and its mechanical properties may be dependent on the type and direction of the stress applied, as well as the prismatic orientation.<sup>5-8</sup>

Between enamel and dentin, a biological interface may dissipate stresses inhibiting further crack propagation.<sup>9,10</sup> The dentin-enamel junction (DEJ) has a high fracture toughness and, along with the more resilient underlying dentin, supports the integrity of enamel by preventing its fracture during function.<sup>11</sup>

Dentin is a hydrated biological composite composed of 70% inorganic material, 18% organic matrix and 12% water (wt%), with properties and structural components that vary with location.<sup>12</sup> The collagen phase of intertubular dentin contributes to a lower modulus of elasticity than enamel, while the lower mineral content is associated with a decrease in dentin microhardness as compared with enamel.<sup>13</sup> The structural composition of dentin includes oriented tubules surrounded by a highly mineralized cuff of peritubular dentin and an intertubular matrix consisting of type I collagen fibrils reinforced with apatite.<sup>14,15</sup> The relative contribution of tubules, peritubular and intertubular dentin varies significantly in composition with location.<sup>16</sup> These differences in composition are thought to have profound effects in its tensile strength.<sup>17</sup>

Due to improvements in the ability of adhesive systems to bond to dental tissues, there has been an increase in the frequency of cohesive failures of dentin during bond strength testing. This precludes the determination of the real interfacial bond strength and has been shown to occur more frequently when conventional shear or tensile methods are used.<sup>18-20</sup>

Since its introduction in 1994, the microtensile technique<sup>5</sup> has proven to be a very useful tool to measure not only the bond strength of adhesive materials to dental tissues, but also to determine the ultimate tensile strength (UTS) of both materials and tooth substrates.<sup>5,17,18,22-26</sup> Major advantages of the microtensile technique include improved stress distribution during adhesion testing<sup>18,27</sup> and the ability to perform the test in very small specimens.<sup>18</sup> Although a reduced number of cohesive failures within the substrate is usually reported when the microtensile technique is used to assess the bond strength of resin materials to dental tissues,<sup>18,21</sup> high-performance adhesive systems

may still induce such cohesive failures in the substrate with this technique.<sup>21,28</sup> Therefore, to facilitate the interpretation of the microtensile bond strength data when failures occur within the substrate, it is important to determine the UTS of the substrate using the same microtensile method.

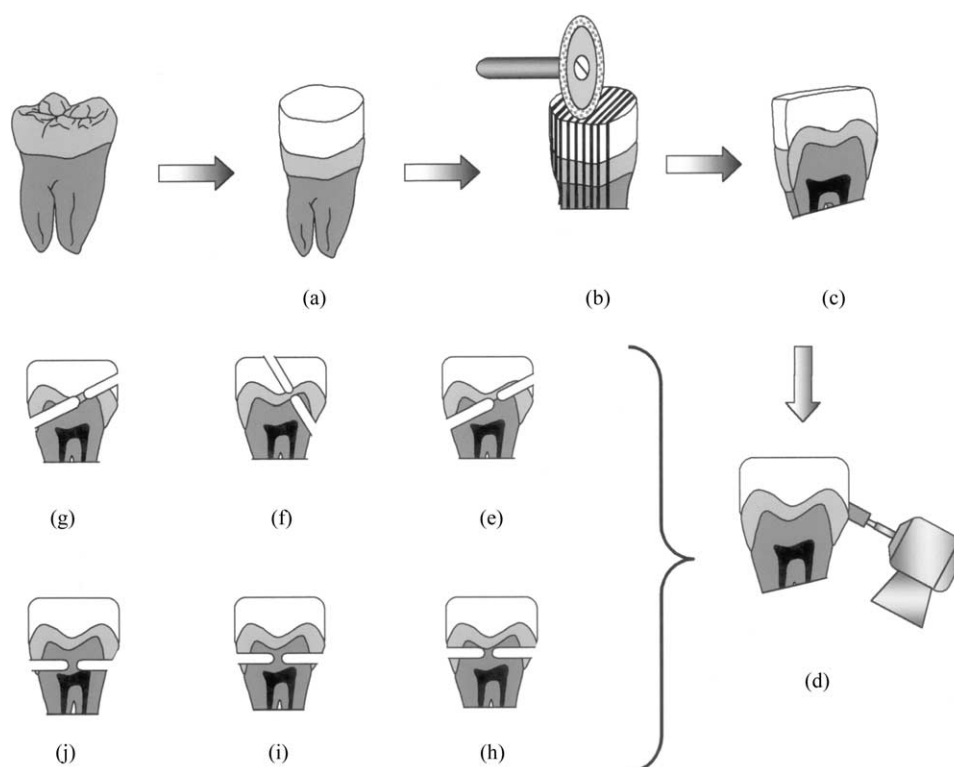
The purpose of this study was to determine the UTS of enamel, dentin and DEJ as a function of their orientation and location. Enamel was tested according to its prismatic orientation, transversally or parallel; the occlusal DEJ was tested; and dentin was tested as function of depth: superficial, middle and deep. In addition, fractured surfaces were examined under a scanning electron microscope (SEM) to determine the failure patterns. Two hypotheses were tested: the first hypothesis was that the apparent UTS of the DEJ is closer to that of enamel than to dentin. The second hypothesis was that deep dentin is weaker than superficial dentin.

## Materials and methods

Twenty sound human third molars that were refrigerated in a solution of 0.05% thymol for no longer than one month after extraction were cleaned of gross debris and placed in distilled water for 24 h before beginning the experiment. The teeth used in this study were obtained under the protocol (75/99) that was analyzed and approved by the Ethical Committee in Research at the Piracicaba Dental School/UNICAMP, Piracicaba, SP, Brazil.

Intact occlusal enamel surface was etched with 37% phosphoric acid for 30 s, rinsed and bonded with single bond (3M ESPE, St Paul, MN, USA). A resin composite block (6 mm high) was incrementally built up in three layers with TPH Spectrum resin composite (Dentsply Caulk, Milford, DE, USA) to form an extension of the crown to facilitate further slicing and testing (Fig. 1a). Each increment was light-cured for 20 s and the specimens were stored in water at 37 °C for 24 h.

The roots were removed approximately 3 mm below the cemento-enamel junction using a diamond disk (KG Sorensen, Barueri, SP, Brazil) mounted in a low-speed handpiece. The remaining crown was then serially, vertically sectioned in a buccal-lingual direction (Fig. 1b) to obtain several slices of approximately 0.7 mm thick (Fig. 1c). Six slices of each tooth were selected and randomly assigned into six groups ( $n = 20$ ). The slices were trimmed to an 'hourglass'-shape with a superfine diamond bur under air-water irrigation (Fig. 1d) to reduce the DEJ (Fig. 1e), enamel or dentin to



**Figure 1** Schematic representation of specimen preparation. (g) Enamel parallel to the prism orientation; (f) enamel stressed perpendicular to enamel prisms. (e) DEJ stressed parallel to enamel prisms; (j) deep dentin was tested; (i) orientation for testing middle dentin parallel to the long axis of the tubules; (h) superficial dentin was tested.

a cross-sectional area of  $0.5 \text{ mm}^2$ . DEJ and enamel specimens were obtained from the internal slope of the buccal cusps. Enamel was trimmed in such a way that permitted the testing by applying the load in an orientation either transversal to its prismatic orientation (Fig. 1f) or parallel to its prismatic orientation (Fig. 1g). Dentin was trimmed as a function of depth: trimming was done 1 mm (superficial), 2 mm (middle) or 3 mm (deep) below the occlusal DEJ (Figs. 1h-j, respectively).

Each specimen was fixed to the grips of the microtensile testing device with cyanoacrylate glue (Zapit, Dental Vent. Am., Corona, CA, USA) and tested in tension at  $0.5 \text{ mm/min}$  in a universal testing machine (4411, Instron Co., Canton, MA, USA) until failure. After fracture, the specimen was removed from the testing apparatus and the cross-sectioned area at the site of fracture measured with a digital caliper (727, Starrett Ind. Com. Ltda., Itu, SP, Brazil) to the nearest  $0.01 \text{ mm}$ . Mean tensile strength values were expressed in MPa and data were analyzed by one-way ANOVA, followed by Duncan's Multiple Range test at  $\alpha = 0.05$ .

The specimens were allowed to air-dry overnight, sputter-coated with gold (MED 010, Balzers, Balzer, Leichtenstein) and examined under an SEM

(DSM 940A, Zeiss, Oberkochen, Germany). Representative areas of the tested sites were photographed at  $2000\times$  and  $5000\times$  magnifications.

## Results

The analysis of variance revealed that there were significant differences among the UTS of the dental substrates ( $p < 0.05$ ). Table 1 shows the average UTS of the substrates. Superficial dentin showed the highest mean tensile strength ( $61.64 \text{ MPa}$ ,  $p < 0.05$ ) of all the substrates. The lowest values were

**Table 1** Ultimate tensile strength (MPa) of tooth structures.

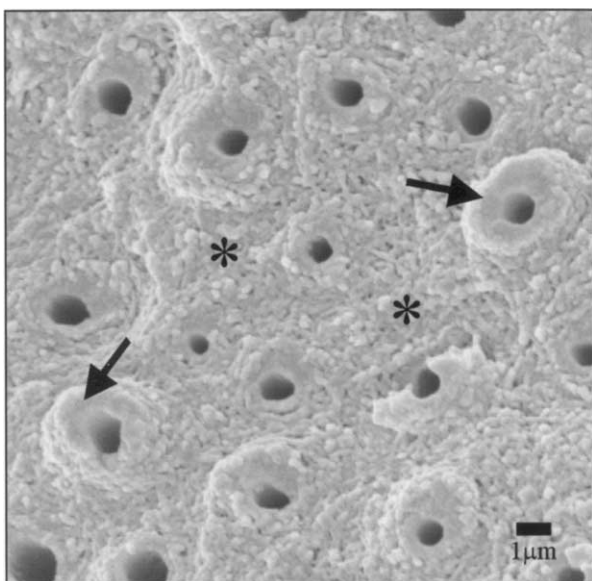
Tooth structure	Mean (SD)
Superficial dentin	61.6 (16.3) <sup>a</sup>
Middle dentin	48.7 (16.7) <sup>b</sup>
Deep dentin	33.9 (8.0) <sup>c</sup>
Dentin-enamel junction	46.9 (13.7) <sup>b</sup>
Parallel enamel	42.2 (12.0) <sup>b</sup>
Transversal enamel	11.5 (4.7) <sup>d</sup>

Means indicated by different letters are significantly different at  $p < 0.05$ ;  $N = 20$  per group.

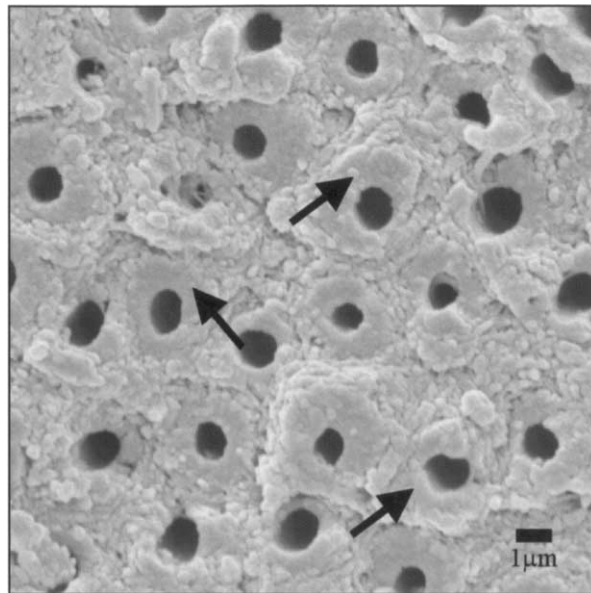
observed for enamel when tested transversally to its prismatic orientation (11.49 MPa,  $p < 0.05$ ). Dentin depth significantly influenced the strength of dentin as it became weaker closer to the pulp ( $p < 0.05$ ). Enamel was significantly stronger when tested parallel to its prismatic orientation ( $p < 0.05$ ). The strength of the DEJ was similar to that of middle dentin and enamel when tested parallel to its prismatic orientation ( $p > 0.05$ ).

Representative SEM micrographs of the fractured site of the specimens are shown in Figs. 2-6. All dentin specimens were fractured across the trimmed area. For superficial dentin, a larger area of solid dentin and low tubule density are noted (Fig. 2). In contrast, a relative increase in the area occupied by tubules and a reduced area of solid dentin are observed in deeper dentin (Fig. 3). In general, fractures did not occur in a sharp, single plane along the intertubular and peritubular dentin. Fractures across the peritubular dentin occurred either above or below the plane of fracture of the intertubular dentin (Figs. 2 and 3).

SEM observation showed that most DEJ specimens actually fractured along the weaker neighboring enamel, thus, intact areas of DEJ can be noted (Fig. 4). When enamel was stressed parallel to its prismatic orientation, fractures occurred preferentially obliquely to the long axis of the prisms, leaving them prominent on the surface. Cone-like ends were generally seen at the fractured site of

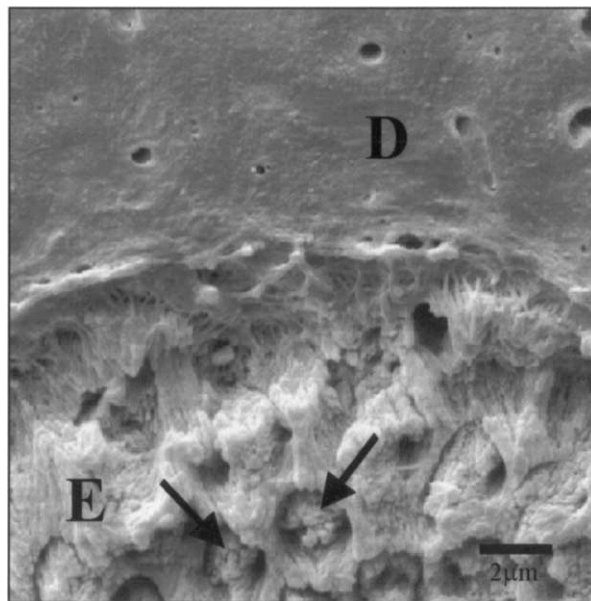


**Figure 2** Fractured site in superficial dentin. Intertubular dentin occupies most of the area (\*). An abrupt change in the plane of fracture was observed at the boundaries between intertubular and peritubular dentin (arrows). Original magnification  $\times 10,000$ .

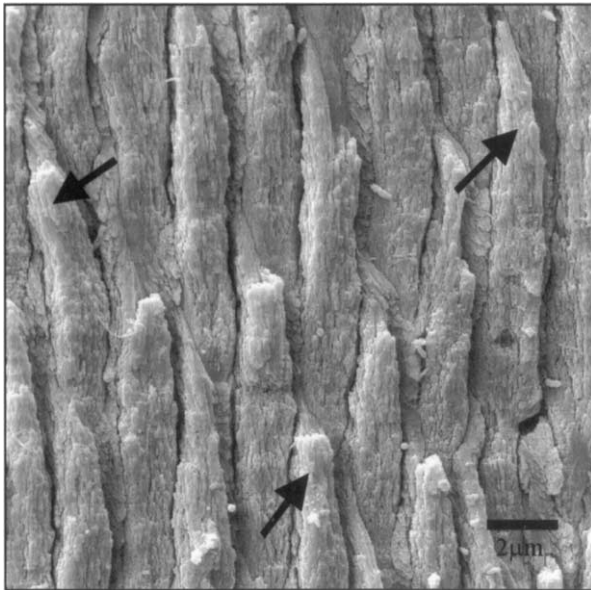


**Figure 3** Fractured site in deep dentin. Tubules are larger in diameter and occupy a relatively larger area of the surface. A similar fracture pattern as for superficial and middle (not shown) dentin was observed. The plane of fracture changes abruptly from intertubular to peritubular dentin (arrows). Original magnification  $\times 10,000$ .

individual prisms (Fig. 5). A characteristic fracture pattern was observed for enamel tested transversally to the orientation of prisms. Fractures occurred preferentially along the interprismatic

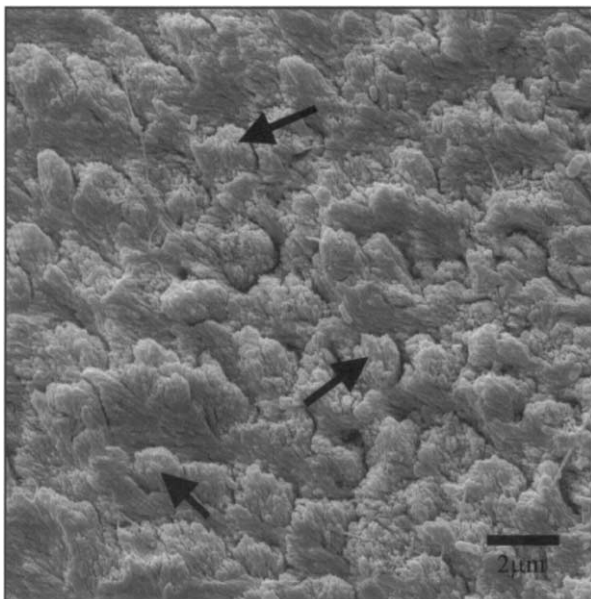


**Figure 4** Fracture site near the dentin-enamel junction. The DEJ is intact as the fracture occurred in the neighboring enamel. At the enamel side (E), typical oblique fractures of enamel prisms can be noted as they were stressed parallel to their orientation (arrows). Dentin (D) adjacent to the DEJ. Original magnification  $\times 5000$ .



**Figure 5** Fractured site of enamel tested parallel to its prismatic orientation. An irregular surface consisting of cone-shaped structures (arrows) was observed. As the fracture did not occur in a single plane, these structures appeared protruding from the surface. Original magnification  $\times 5000$ .

substance and parallel to the long axis of the prisms. Oblique fractures across the prisms could be noted at sites where the prism changes its orientation (Fig. 6).



**Figure 6** Fractured site of enamel tested transversally to its prismatic orientation. Fracture plane occurred preferentially along the interprismatic substance. Whenever prisms changed their orientation, oblique fractures of the prisms were noted (arrows). Original magnification  $\times 5000$ .

## Discussion

Bond strength testing methods are designed to determine the interfacial strength between the bonded substrates. However, in such testing, the applied load cannot be exclusively concentrated at the interface, but it is rather distributed and modified along the bonded substrates, depending on the composition of the substrates and the geometry of the testing assembly.<sup>19,20</sup> Therefore, the intrinsic strength of the bonded substrates can act as a modifier of the resultant apparent bond strength value.<sup>28-30</sup> When testing resin bond strength to dentin using conventional shear testing, cohesive failures within the substrate have been reported to occur in the range of 20-30 MPa.<sup>18</sup> Because the intrinsic cohesive strength of dentin has been reported to be much higher (ca. 50-130 MPa),<sup>11,17,22,31,32</sup> fractures below those values are generally confusing and attributed to other factors related to the testing method.<sup>19,20,29</sup> Gwinnett<sup>33</sup> demonstrated that the cohesive strength of dentin was about 30-35 MPa when tested in specimens with a similar geometry to that used with the shear bond strength method, indicating a more realistic value for that specific testing method.

Over the last 5 years, there have been an increasing number of studies using the microtensile technique to evaluate the bond strength of adhesive materials to dental tissues. This method tends to decrease the number of pure cohesive failures within the substrates compared to conventional testing techniques.<sup>18,34</sup> However, since cohesive failures of dentin still occur, it is important to determine the strength of the dental structures when tested using the similar microtensile technique.

Most of the tooth structure is comprised of dentin. The tubule density and the area occupied by solid dentin vary with distance from the pulp chamber to the DEJ.<sup>35-37</sup> Several studies have investigated the relationship between microstructure and the mechanical properties of dentin.<sup>1,17,22,23,31,38,39</sup> Results indicated that cohesive strength of dentin varies significantly and is dependent on intra-tooth location.<sup>6,24,32,38,40</sup> In this study, statistically significant differences were found in the UTS of dentin as function of depth. Our results confirm that dentin becomes weaker closer to the pulp than near the DEJ, as previously demonstrated by others.<sup>17,24,32,38,39</sup> These results support the second hypothesis that deep dentin is weaker than superficial dentin. It is evident that the empty lumina of the tubules do not

contribute to the strength of dentin, therefore, the relative larger area of solid dentin in superficial rather than in middle and deep dentin explains the findings. Our values are within the same range as those previously reported for specimens originating from the same regions in dentin and tested under similar methods.<sup>17,24</sup> Sano et al.<sup>22</sup> reported higher UTS values for dentin when the tensile force was directed perpendicular to the tubule orientation. Further, their specimens had a smaller cross-sectional area (ca. 0.25 mm<sup>2</sup>) and that is known to result in apparent higher values of either interfacial or cohesive strength of materials.<sup>17,21</sup> Our specimens were tested with the load applied parallel to the orientation of the tubules. Apparently, dentin is stronger when the load is applied transversally to that orientation.<sup>17</sup> Since specimens for bond strength testing are generally prepared on a transversally flattened dentin surface, usually located in superficial or middle dentin, our results indicate that bond strength values above 45 MPa may generate cohesive failures of dentin when using the microtensile technique.

The DEJ is a unique junctional zone between highly mineralized tissues of different embryogenic origins, matrix composition and physical properties. SEM investigations of DEJ surfaces showed that both enamel and dentin are scalloped in nature.<sup>41,42</sup> None of the specimens tested in this study fractured solely along the DEJ. Fractures occurred more frequently in enamel and cracks always propagated following the orientation of the prisms. Thus, the results support the first hypothesis that the apparent UTS of the DEJ is closer to enamel than to superficial dentin. It is interesting to note that the average UTS of the DEJ was not significantly different from the UTS of enamel when stressed parallel to its prismatic orientation (Table 1). By the way we prepared our enamel specimens, the orientation of the enamel prisms just below the DEJ was parallel to the enamel prisms (Fig. 1e). Similar fracture patterns and strength values were obtained by recent studies on the physical properties of the DEJ.<sup>5,43</sup> The lack of pure interfacial failure can be attributed to the complexity of DEJ structure and its ability to modify crack propagation.<sup>5,8</sup> It must be emphasized that the 'DEJ' values do not represent the UTS of the DEJ, but that of nearby enamel. The toughness of the DEJ precluded attempts to measure its UTS.

Previous studies on the physical properties of enamel have considered it as a brittle structure with anisotropic behavior.<sup>2,6-8,25</sup> Crack propagation analysis usually demonstrates that enamel fractures occur preferentially along the weakest path,

i.e. around or between prisms, which behave as integral units and are less likely to cleave under tension.<sup>6-8</sup> A significantly lower load was able to fracture enamel when it was loaded in a direction perpendicular to the orientation of the prisms (Table 1). This is similar to the results of other studies.<sup>7,25,28</sup> In that situation, the weaker interprismatic substance is also aligned perpendicular to the load and rapidly propagates the tension across the specimen causing it to fail under a lower tension. In such a case, enamel prisms are separated from one another along the interprismatic substance and only a few are obliquely cleaved. When enamel is stressed parallel to its prismatic orientation, the tension concentrates on the stronger prismatic units and the fracture requires that all the prisms be cleaved before catastrophic failure can occur. This results in significantly higher UTS for enamel when tested under this condition (Table 1). Figs. 5 and 6 show the fracture pattern of both orientations and are consistent with previous reports.<sup>6,25</sup> The mechanical behavior of enamel has profound relevance for clinical bonding procedures.<sup>25</sup> Depending on the location and treatment of enamel margins in a cavity, factors such as the presence of a bevel, contraction stresses generated during the polymerization of the resin and the moment of finishing and polishing, enamel cracks may occur and ultimately compromise the clinical success of the restorative procedure.<sup>21,28,44,45</sup> Enamel butt joint margins at both occlusal and cervical areas are more likely subjected to stresses produced by restorative procedures<sup>45</sup> and the need for placement of a bevel at such margins has been recently questioned.<sup>21,28</sup>

The results of this study indicate that the UTS of dentin, the DEJ and enamel vary widely. Additionally, enamel showed an anisotropic behavior and dentin strength was influenced by intra-tooth location. Perhaps deep dentin is weaker because it has larger, more numerous tubules and that these tubules might serve as sites for crack initiation.<sup>38</sup> The study provides data on the mechanical properties of such structures that were tested under conditions similar to those when using the microtensile bond strength method. Such information may be useful for a better understanding and interpretation of bond strength data obtained using microtensile method.

## Conclusion

The results of this study confirm that the UTS of tooth structures is dependent on intra-tooth

location and the nature of the substrate. Enamel and dentin tensile properties are influenced by prismatic orientation and distance from the DEJ, respectively.

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