



## Short communication

## Importance of tubule density to the fracture toughness of dentin

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## ABSTRACT

**Objective:** The fracture toughness of dentin is critical to the prevention of tooth fracture. Within the tooth crown, the mechanical properties of dentin are influenced by spatial variations in the density and diameter of the dentin tubules with distance from the pulp. There are also relevant changes to the microstructure of dentin with age. In this investigation the importance of tubule density to the fracture toughness of dentin was evaluated in “young” and “old” age groups.

**Methods:** The variations in microstructure (density and diameter of tubules) from young and old donor teeth were studied by means of optical microscopy.

**Results:** A reduction in the density and diameter of tubules was identified to occur with aging. An approach previously proposed to study the mechanical behavior of porous materials was used to model the fracture toughness of coronal dentin in terms of the tubule characteristics. Results were then compared with published results from previous studies.

**Conclusions:** The model predictions were consistent with experimental results for the fracture toughness of dentin from young donor teeth, but overestimated the values that have been reported for “old” dentin.

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## 1. Introduction

The “cracked tooth syndrome” was coined by Cameron (1964) over a half-century ago to describe the prominence of cusp fractures observed in restored teeth. Fracture is no less important to the field of restorative dentistry today (e.g. Barreto et al., 2015; Opdam et al., 2014).

The human dentition is subjected to a variety of cyclic stresses, including those of mastication and other *para*-functional activities. Acute stresses can promote the formation of microcracks in the tooth (Homewood, 1998), in addition to other events including trauma and restorative procedures (Bastone, Freer, & McNamara, 2000; Lee et al., 2014; Majd, Viray, Porter, Romberg, & Arola, 2012). Damage and microcracks in dentin may not cause fracture, but can facilitate the failure of restored teeth through fatigue crack growth (Arola, Huang, & Sultan, 1999; Bajaj, Sundaram, & Arola, 2008; Nalla, Imbeni et al., 2003). Spatial variations in the resistance to fracture of dentin can contribute to this process and is an important factor to consider.

Due to its importance, the fracture toughness of coronal dentin has been evaluated, with primary emphasis on the importance of

tubule orientation. For instance, El Mowafy and Watts (1986) measured the fracture toughness of dentin for cracks oriented parallel to the dentinal tubules. The average fracture toughness reported was 3.08 MPa m<sup>0.5</sup>, with no dependence of temperature between 0 °C and 60 °C. Imbeni, Nalla, Bosi, Kinney, and Ritchie (2003) employed a three-point bending approach to achieve crack extension perpendicular to the long axis of the tubules, and reported an average fracture toughness of 1.8 MPa m<sup>0.5</sup>. Further, Iwamoto and Ruse (2003) measured the fracture toughness of dentin for three different orientations relative to the dentinal tubules, including directions denoted perpendicular, parallel, and parallel-transverse. No significant difference was found between the parallel (1.97 MPa m<sup>0.5</sup>) and parallel-transverse (2.02 MPa m<sup>0.5</sup>) orientations. However, the average value for the perpendicular orientation (1.13 MPa m<sup>0.5</sup>) was significantly lower.

With the increase in senior dentate, aging has become of greater importance to the field of restorative dentistry (McNally, Matthews, Clovis, Brilliant, & Filiaggi, 2014; Yellowitz & Schneiderman, 2014). Human teeth undergo changes with increasing age, including a decrease in the number of odontoblasts, an increase in dentin thickness and the formation of transparent dentin (Bernick & Nedelman, 1975; Murray, Stanley, Matthews, Sloan, & Smith, 2002; Nanci, 2012; Timiras, 2007; Toto, Kastelic, Duyvejonck, & Rapp, 1971). In addition, there are changes in mechanical properties, such as an increase in elastic modulus and hardness (Senawongse, Otsuki, Tagami, & Mjor, 2006), a decrease in strength

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(Arola & Reprogel, 2005; Kinney, Nalla, Pople, Breunig, & Ritchie, 2005) and decrease in fatigue crack growth resistance (Bajaj, Sundaram, Nazari, & Arola, 2006). There is also a reduction in fracture toughness with increasing age. Kinney et al. (2005) reported that the fracture toughness of transparent dentin, after nearly complete filling of the tubules by sclerosis, was  $1.46 \text{ MPa m}^{0.5}$  and significantly lower than that for young dentin. The decrease in toughness appears to be independent of tubule orientation (Koester, Ager, & Ritchie, 2008b; Nazari, Bajaj, Zhang, Romberg, & Arola, 2009). Changes in the fracture toughness with aging may be related to dehydration (Toto et al., 1971), increase in the degree of mineralization (Porter et al., 2005), or an increase in the degree of crosslinking of the collagen fibrils (Miura et al., 2014). However, the most visible change is the decrease in diameter and density of the tubules, which results from an increase in mineral deposited within the tubules.

To the authors' knowledge, no investigation has examined the fracture toughness of coronal dentin in terms of tubule characteristics and number of obliterated tubules that occur with increasing age. Hence, the objective of this investigation was to examine the effect of tubule density on the fracture toughness of dentin from young and old donors and utilize an approach previously proposed for porous materials to quantify the spatial variations in fracture resistance.

## 2. Materials and methods

Human third molars were obtained from selected patients after written consent and following all the protocols required by the Dental Clinic at Universidad Cooperativa de Colombia (UCC). Exclusion criteria included presence of caries and previous restorations. The teeth were obtained from donors residing in Medellín, Colombia, and were divided into two age groups, namely a "young" group with donors between 18 and 25 years of age ( $N=12$ ), and an "old" group with donors between 47 and 65 years of age ( $N=8$ ). There were an equal number of male and female samples in both groups. Immediately after extraction, all the specimens were kept in Hank's Balanced Salt Solution (HBSS) at  $2^\circ\text{C}$  to prevent dehydration (Habelitz, Marshall, Marshall, & Balooch, 2001). In addition, the specimens were tested within two weeks of extraction to limit the loss of mineral and potential degradation of organic materials.

Each molar was sectioned along its longitudinal axis (section A–A in Fig. 1a) using diamond abrasive slicing equipment with continuous water coolant. Secondary sections were made

transversely (section A'–A') in order to expose the dentin (Fig. 1b). For microscopic evaluations the specimens were embedded in cold-cured epoxy resin and then polished using silicon carbide abrasive paper with successively smaller particle sizes until reaching #1200 grit. Further polishing was then performed using diamond particle suspensions ( $3 \mu\text{m}$  particles) with standard red felt polishing cloth wheels. The polished specimens were then kept in a HBSS bath solution.

The dentin sections were evaluated using optical microscopy (Axiovert 40 MAT, Carl Zeiss Microscopy, NY) to characterize the microstructure. Tubule density and tubule lumen diameter were measured within the regions corresponding to outer, middle and inner dentin. These measurements were located approximately at 0.5 mm, 2.0 mm and 3.5 mm away from the dentinal enamel junction (DEJ), respectively.

Measurement of the tubule density ( $\rho_t$ ) and tubule lumen diameter ( $\phi_l$ ) was performed using commercial image analysis software (AxioVision LE). Seven randomly selected images with a constant area (approximate size of each image  $80 \mu\text{m} \times 100 \mu\text{m}$ ) were obtained over the polished surface. The mean tubule diameter and number of tubules were calculated for each image. Values from the seven images were averaged and used to estimate the lumen area fraction ( $\xi$ ) within the three regions of evaluation as

$$\xi = \frac{A_l}{A_T} \quad (1)$$

where  $A_l$  is the area ( $\text{mm}^2$ ) occupied by lumens and  $A_T$  is the total area of dentin measured ( $\text{mm}^2$ ) in each image. The average lumen area was calculated using the measures of tubule diameter and density.

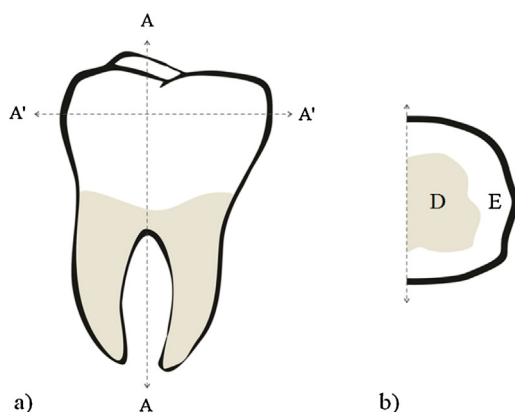
## 3. Results

The microstructure of dentin from selected young and old donor teeth evaluated is shown in Fig. 2. Representative images are presented from the three regions of evaluation including the outer (Fig. 2a and b), middle (Fig. 2c and d) and inner (Fig. 2e and f) dentin. For both age groups the peritubular dentin can be seen surrounding each dentinal tubule. Several obliterated tubules are evident in the images for the old donor group, with greater number of filled tubules in the middle and outer regions. Micrographs of tubules within the outer dentin of young and old donor teeth obtained at higher magnification are shown in Fig. 3(a) and (b), respectively. An example of a tubule that has become filled with mineral is shown in Fig. 3(b). No obliterated tubules were evident in the dentin of the young donor group regardless of the region of evaluation.

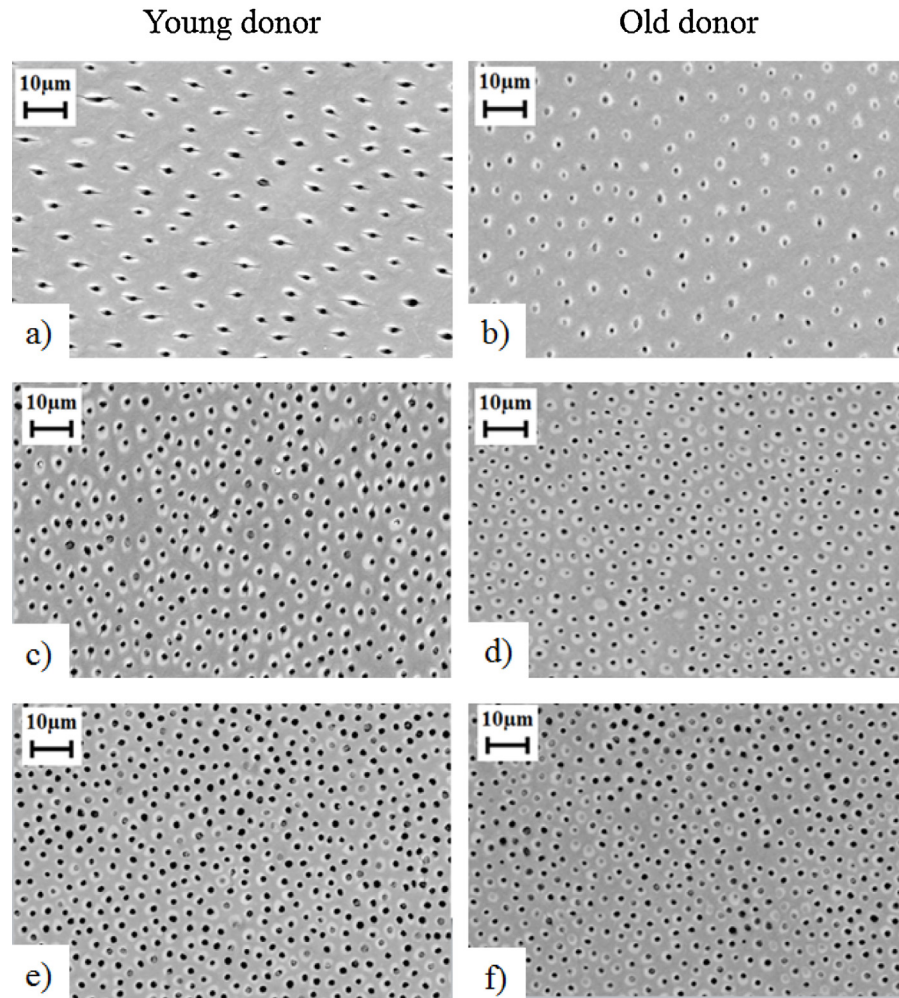
Fig. 4 shows the estimates for the lumen area fractions ( $\xi$ ) in the three regions of dentin evaluated. Overall, there was a significant decrease in  $\xi$  with proximity to the DEJ for both age groups ( $p \leq 0.05$ ). In the young donor group the average  $\xi$  in the outer and inner regions was  $3.7 \pm 0.6\%$  and  $9.3 \pm 1.0\%$ , respectively. For the old group these values were  $2.9 \pm 0.4\%$  and  $9.6 \pm 1.1\%$ , respectively. The primary difference between the two age groups was the lower value of  $\xi$  (37% less) in the outer region of dentin for the old group. This difference is attributed to the reduction in diameter of the dentin tubules due to deposition of mineral within the lumens as a result of sclerosis. When comparing the results for  $\xi$  in each region between the young and old donors, significant differences were found only for outer dentin ( $p \leq 0.05$ ).

## 4. Discussion

While the dentin tubules play an important role in tooth sensitivity and pain stimuli (Magloire et al., 2010), they also serve



**Fig. 1.** Schematic diagram of a sectioned molar after (a) longitudinal (A–A), and (b) transverse (A'–A') cutting. After sectioning the specimens, they were embedded in cold-cure epoxy resin with the sectioned surface facing outwards. The letters D and E refer to dentin and enamel, respectively.



**Fig. 2.** Micrographs of the microstructure for the young and old dentin as a function of location. (a and b) Outer dentin; (c and d) middle dentin; (e and f) inner dentin. Note the regions of obliterated dentinal tubules in micrographs for the middle and outer dentin of the old donor teeth.

as a form of porosity that contributes to the mechanical behavior of dentin. Several models have been proposed to describe the mechanical behavior of porous materials (Hasselman, 1963; Ji, Gu, & Xia, 2006; Ryshkewitch, 1953; Schiller, 1985). According to these models, it is possible to define relationships between porosity and physical properties such as Young's modulus, hardness, and strength (Ji et al., 2006; Kováčik, 1999; Luo & Stevens, 1999; Salomon & Kosmač, 2013). However, the lack of uniformity in the distribution and shape of pores has been considered a limitation of these approaches in their general application to materials.

Balshin (1949) developed an empirical model for the prediction of mechanical properties for materials with microstructures similar to dentin. According to this approach, the variation in fracture toughness of dentin can be described as a function of the porosity or the lumen area fraction ( $\xi$ ) by

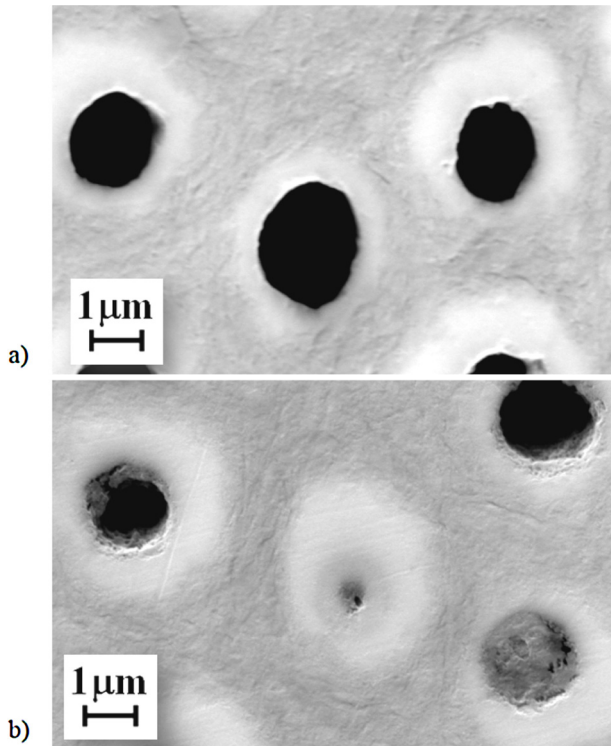
$$K_{ic} = K_{ic_0} (1 - \xi)^m, \quad (2)$$

where  $K_{ic}$  and  $K_{ic_0}$  are the Mode I fracture toughness of dentin with specific porosity and of solid dentin (i.e. with no lumens), respectively. The exponent  $m$  is a constant that depends on the degree of stress concentration developed around the lumens (i.e. lumen geometry). According to Boccaccini et al. (1996) the value of  $m$  varies from 1, for long cylindrical pores orientated parallel to the

stress direction ( $z/x \sim \infty$ ), up to about 7 for oblate spheroids (axial ratio  $z/x < 1$ ). The axial ratio  $z/x$  is related to the shape of the pores, where  $z$  and  $x$  are associated with the length and width of the pore, respectively.

The reported range in fracture toughness for human dentin is roughly  $1.1 \text{ MPa m}^{0.5} \leq K_{ic} \leq 3.5 \text{ MPa m}^{0.5}$ . The lowest values have been obtained for cracks oriented perpendicular to the tubules (Iwamoto & Ruse, 2003), whereas higher values are reported for cracks extending in-plane with the tubules (Yan, Taskanak, & Mecholsky, 2009) and for outer dentin, which exhibits a low tubule density (Pashley, Okabe, & Parham, 1985). Nonetheless, estimates for the fracture toughness of dentin without the presence of dentinal tubules (solid dentin) have not been reported.

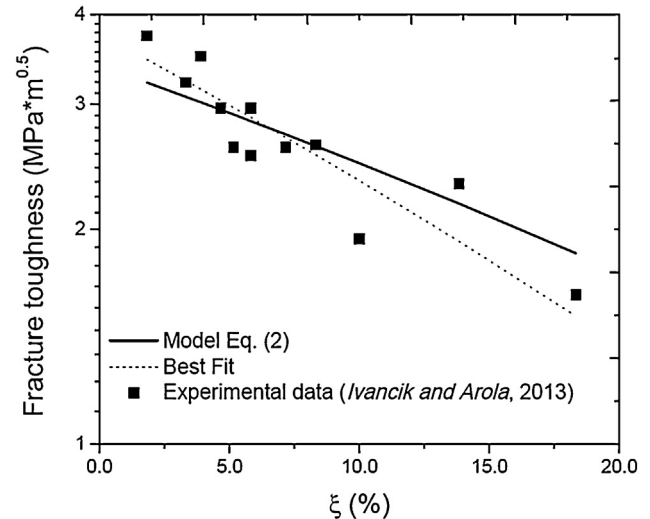
Recently, Ivancik and Arola (2013) evaluated the fracture behavior of coronal dentin obtained from the teeth of young donors residing in the US. They obtained estimates of  $K_{ic}$  for the outer, middle, and inner dentin. The average fracture toughness for the outer coronal dentin (with lowest density of tubules) was  $3.40 \text{ MPa m}^{0.5}$ . Their results are shown in Fig. 5 along with a prediction from the Balshin model (Eq. (2)) using  $K_{ic_0} = 3.40 \text{ MPa m}^{0.5}$  (from outer dentin) and  $m = 3$ , which is appropriate for long cylindrical pores (e.g. dentinal tubules) oriented perpendicular to the direction of stress (Boccaccini et al., 1996). As evident in Fig. 5, the model predicts a decrease in fracture toughness of dentin with



**Fig. 3.** Micrographs of dentinal tubules from the outer dentin of teeth from representative (a) young and (b) old donors.

increasing tubule area fraction (porosity). Interestingly, the distribution correctly captures the decrease in fracture toughness approaching the pulp due to a higher proportion of dentin tubules and larger average diameter. A reasonable correlation was found between the model and experimental results ( $R^2 = 0.89$ ).

An alternate approach was also used to obtain the parameters of Eq. (2) by performing a best-fit to the experimental results (Fig. 5), which resulted in  $K_{ic0} = 3.76 \text{ MPa m}^{0.5}$  and  $m = 4.5$ . A comparison of the model to experimental data with these “best-fit” parameters results in  $R^2 = 0.99$ , which indicates very good agreement. The value obtained for  $K_{ic0}$  is similar to the maximum value obtained by Ivancik and Arola (2013) of  $3.74 \text{ MPa m}^{0.5}$ , which was obtained for the outer dentin with a lumen area fraction of just under 2%. The value of  $K_{ic0}$  estimated in this study is effectively related to dentin

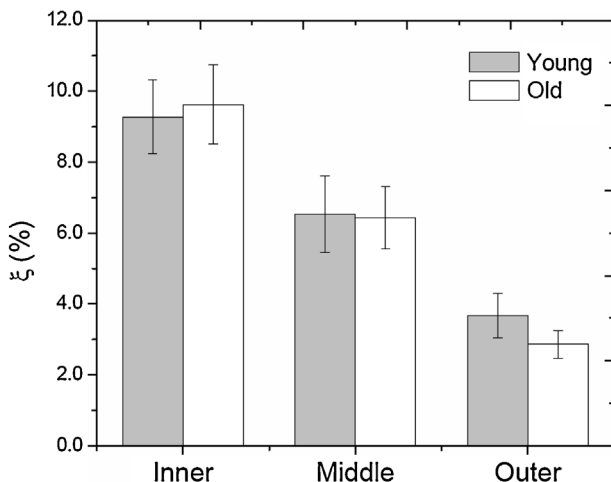


**Fig. 5.** Comparison of experimental and predicted fracture toughness of dentin as a function of the lumen area fraction ( $\xi$ ). The experimental data corresponds to Ivancik and Arola (2013).

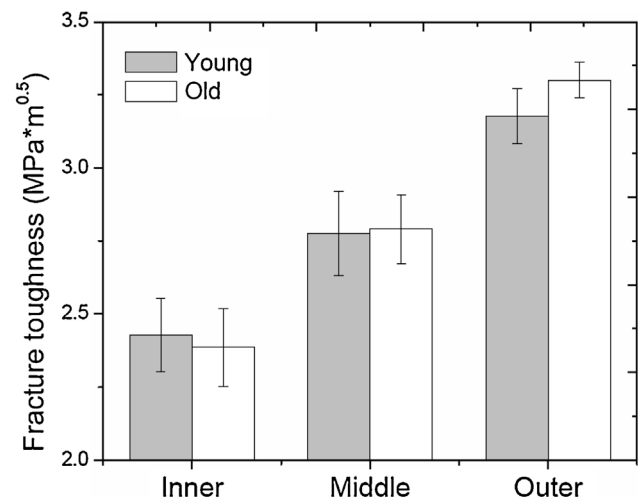
without tubules. According to Boccaccini, Ondracek, and Mombello (1996), a value of  $m = 4.5$  corresponds to a porous microstructure composed of oblate spheroids with  $z/x = 0.3$  and an orientation of  $\alpha = 30^\circ$ , which is a reasonable approximation to the mean shape and orientation of the S-shaped lumens in dentin. Therefore, the model provides a reasonable description for the effect of  $\xi$  on the fracture toughness of coronal dentin.

Estimates for the fracture toughness of dentin from the teeth of young and old Colombian donors were obtained using Eq. (2) and are shown in Fig. 6. These predictions were obtained using the best-fit estimates for  $K_{ic0}$  and  $m$ , along with the lumen area fraction measurements ( $\xi$ ) from the Colombian donor teeth in Fig. 4. As evident in this figure, there is a significant increase ( $p \leq 0.05$ ) in fracture toughness with increasing proximity to the DEJ for the middle and outer regions of young dentin, which agrees with the reported results (Ivancik & Arola, 2013).

Furthermore, in comparing the estimated  $K_{ic}$  between the young and old dentin of the three regions there is a significant difference ( $p \leq 0.05$ ) in values for the outer dentin only; the  $K_{ic}$  for



**Fig. 4.** Lumen area fraction ( $\xi$ ) for the three different regions of coronal dentin and a comparison of results for the young and old donor teeth.



**Fig. 6.** Estimated fracture toughness for different regions of coronal dentin for the young and old donor teeth. These estimates are obtained from the lumen area fraction ( $\xi$ ) measurements and the use of the Balshin equation (Balshin, 1949).



the old dentin is 4.0% greater. These results contrast those previously reported for the fracture toughness of dentin from senior patients (Kinney et al., 2005; Nazari et al., 2009) where a substantial reduction (20%) in fracture toughness was noted with aging, regardless of the direction of crack growth (Ivancik & Arola, 2013; Koester, Ager, & Ritchie, 2008a).

According to the adopted model (Eq. (2)), the microstructural changes in dentin that occur with aging would cause an increase in fracture toughness. This behavior is related to the reduction in number and diameter of stress concentrators in the material (as represented by lower  $\xi$ ). But if the tubules are obliterated by deposited mineral with low fracture toughness, the overall toughness should be higher than that of a porous material (with empty tubules) if the mechanisms of fracture do not change with aging. An obvious limitation of the proposed model is that only geometrical features are considered (i.e. lumen area). The question that arises is “If the dentin of old patients undergoes a decrease in the number of stress concentrators as a result of sclerosis, why is there a reduction in the fracture toughness with aging?”

The primary mechanisms of crack growth toughening in dentin involve crack deflection, uncracked-ligament bridging, crack branching and collagen fibril bridging (Nalla, Kinney, & Ritchie, 2003; Nalla, Kinney, Tomsia, & Ritchie, 2006). *In situ* observations of crack growth in dentin have shown that the obliteration of tubules in old dentin causes changes to the path of crack growth (Koester et al., 2008b) and the mechanisms of toughening. Specifically, tubules that are filled with mineral resist penetration of the crack. Consequently, crack extension occurs about the interface of the peritubular cuff and intertubular dentin. In young dentin, the peritubular cuffs located within the K-dominant region undergo fracture. As a consequence, crack growth occurs by the “linking” of these spurious microcracked tubules or so-called daughter cracks (Ivancik, Majd, Bajaj, Romberg, & Arola, 2012; Nalla et al., 2006). Due to the suppression of peritubular cuff fracture in old dentin, crack deflection and uncracked-ligament bridging (two of the dominant mechanisms of toughening) are essentially inactivated. Koester et al. (2008b) commented that the most commonly observed change in fracture characteristics with aging was a decrease in crack branching. That process occurs by crack extension from tubule to tubule and the propensity for branching is determined by the number of unfilled tubules. Therefore, aging appears to cause a decrease in fracture toughness of dentin due to a reduction in the number of contributing toughening mechanisms as well as a potential reduction in the potency of remaining mechanisms. A definitive description for the changes in toughening mechanisms in dentin with aging has not been presented. A quantitative description for the reduction in toughness related to the individual mechanisms, including the increase in mineralization of intertubular dentin, cross-linking or degradation of the collagen matrix, would be valuable.

The changes in toughening mechanisms with aging necessitates that new values of  $K_{IC}$  and  $m$  are obtained for the model (Eq. (2)) if applied to old dentin. Some modifications in the Balshin model parameters may also be required due to differences in the fracture resistance of dentin between the teeth of US and Colombian donors. Ivancik et al. (2014) compared the microstructure and fracture resistance of dentin from US and Colombian donor teeth. Results showed that there were significant differences in the tubule lumen diameters between the two groups in the inner and outer dentin. Furthermore, it was found that the fatigue crack growth resistance for the dentin of teeth from the Colombian donors was independent of location, which contradicts observed trends in the dentin of US donors. Thus, if the Balshin model is adopted to describe the fracture behavior of dentin, the value of  $K_{IC}$  must be tuned to the unique physical characteristics and the degree of mineralization for the tissue of that patient group.

In past studies the reported reduction in fracture toughness of dentin with age has been attributed to a transformation in the toughening mechanisms. Clearly that results from the changes in microstructure of dentin with increasing patient age. While the most obvious change in microstructure is filling of the lumens with mineral, there are others to consider as well. For example, there are spatial variations in the mineral and collagen contents in the crown (Ryou et al., 2011; Tesch et al., 2001), which changes as a result of the aging process. In addition, there are distinct differences in the degree of mineralization between intertubular and peritubular dentin (Gómez de Ferraris & Campos Muñoz, 2009), which results in a well-defined boundary. It is unclear if this interface undergoes any changes with aging. Another relevant feature of the microstructure to consider is the branching of dentinal tubules (Goracci & Mori, 1995; Szabó, Trombitás, & Szabó, 1984). The tubule branches also undergo filling with mineral, which is a less-recognized result of the aging process. That could contribute substantially to the toughness, but has not been considered in previous evaluations. These factors are also not reflected in the current format of the Balshin model. To obtain a better fit of the model, it would be valuable to identify how these factors affect the mechanical behavior of dentin in both young and old donors and how they could be included as additional parameters of the model.

## 5. Conclusions

On the basis of the results obtained, the following conclusions may be drawn:

1. A quantitative analysis of the changes in tubules occurring in coronal dentin with aging showed that the primary difference between young and old dentin was a reduction in the lumen area fraction. In the outer dentin there was nearly a 40% reduction in the lumen area with aging. This difference is attributed to obliteration of dentinal tubules with formation of sclerotic dentin.
2. An approach previously proposed to study the mechanical behavior of porous materials was employed to model the fracture toughness of dentin and the influence of spatial variations in dentinal tubules. The model showed a strong correlation to experimental results previously reported in the literature for the dentin of young donor teeth. However, results for the old dentin were not in agreement due to differences in the mechanisms of fracture.

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