A micro-mechanics model of dentin mechanical properties

Qing-Hua Qin\textsuperscript{a,b,\#,1}, Michael V. Swain\textsuperscript{b,c}

\textsuperscript{a} Department of Mechanics, Tianjin University, Tianjin 300072, China
\textsuperscript{b} School of Aerospace, Mechanical, Mechatronic Engineering, University of Sydney, Sydney NSW 2006, Australia
\textsuperscript{c} Biomaterials Science Research Unit, Faculty of Dentistry, University, Sydney NSW 2006, Australia

Received 31 July 2003; accepted 4 December 2003

Abstract

Application of a micro-mechanics cell model to dentin composites for determination of their effective mechanical properties is discussed in this paper. The dilute micro-mechanics model for fibre-reinforced composites is utilized and the corresponding cell model is chosen to consist of a circular hollow cylinder filled with liquid or gas phase, which is surrounded by two circular cylindrical shells, a thin shell and a matrix phase. Each layer of cylindrical shell is here considered as a composite consisting of collagen fibrils, with mineralized hydroxyapatite, loosely connected to their neighbours, and water (or gas in the case of dry dentin composite). Determination of the effective material properties of such a three phase composite is discussed. Using the cell model the effect of porosity, thickness of each cylindrical shell, and mineral content on material properties is analysed. Results obtained from nano-indentation observations are compared with numerical predictions of the analytical model.

\textcopyright{} 2004 Elsevier Ltd. All rights reserved.

Keywords: Dentine; Mechanical properties; Collagen; Constitutive modelling

1. Introduction

Recently, considerable attention has been directed toward dentin-like composites with interpenetrating or multiple phases and porosity. In contrast, most conventional composites in which only the matrix phase is spatially continuous and no porosity or liquid phase is involved, dentin is a porous interpenetrating phase composite that has several phases such as collagen fibrils, mineralized hydroxyapatite and water that are each interconnected in three dimensions and construct a topologically continuous network throughout the composite \cite{1,2}. It is recognized that dentin micro-structure and properties are principal determinants of nearly all surgical procedures in preventive and restorative dentistry. Therefore, the determination of the effective mechanical properties of dentin, a multiphase composite, is of great interest for both its biomedical engineering applications and theoretical analysis. However, there has been little previous theoretical work on this specific subject.

The limited size and shape complexity of teeth result in hardness of teeth being the major method of mechanical property characterization. Hardness of teeth has been evaluated by indentation \cite{3,4}. Craig and Peyton \cite{3} examined the difference between hardness of different types of teeth, such as molars and incisors. They found that there was no clear trend in hardness between different areas of the same tooth. The use of indentation to characterize the elastic modulus of teeth was demonstrated by Willems et al. \cite{4}. Xu et al. \cite{5} investigated the crack propagation behaviour and the mechanical properties of teeth on a microscopic scale by means of an indentation method. Currey \cite{6} examined the effects of porosity and mineral content on Young’s modulus of compact bone. It was found that an increase in the mineral content of bone should, other things like porosity being equal, produce an increase in the value of Young’s modulus. It was also confirmed that the third power law relationship between modulus and mineral content presented in \cite{7} could best fit the data obtained. As for the application of micro-mechanical models to dentin materials, studies in \cite{8,9} attempted to model the elastic properties of hard tissues, which focused on the constituent materials properties at the microscopic level.
by means of bounding principles including Voigt and Reuss as well as Hashin–Shtrikman. Gottesman and Hashin [10] developed a micro-mechanics model capable of analysing viscoelastic properties of bone based on a composite cylinder assemblage model which was initially developed for fibre composites [11]. Jones and Boyde [12] described in detail the main feature of dentin microstructure. Kinney et al. [13] studied the effect of tubule orientation on the elastic properties of dentin by way of a micro-mechanics model of cylindrical inclusions in a homogeneous matrix phase [14]. More recently, Kinney et al. [15] presented a critical review on mechanical properties of human dentin composites and indicated that the elastic properties are hexagonal, with the stiff direction perpendicular to the tubules, the precise opposite of the micro-mechanics predictions. None of the existing models, however, takes into consideration the actual detailed micro-structure of dentin composites, as described by the experiments of recent years [5,16,17].

The purpose of this paper is to extend the micro-mechanics models presented in [11,18,19] to the case of fully or partially saturated dentin composites. It is noted that the inclusion and matrix phases are themselves also three phase composites containing collagen fibrils, mineralized hydroxyapatite and liquid phase such as water or gas. We have proposed a micro-mechanics approach to handle this category of composites. Numerical examples are considered that show the applicability of the proposed micro-mechanical model and to identify the effect of porosity, thickness of each cylindrical shell, mineral content, and degree of hydration saturation on material properties. These predictions are compared with previously results reported in [13].

2. Effective properties of peri-tubule inclusion and bonding matrix

The accurate micro-mechanical modelling of actual dentin porous materials is very complicated due to its irregular micro-structural nature and the geometries of real porous material structures. Dentin is a composite material, consisting of peri-tubule inclusion, bonding matrix and water. The peri-tubular inclusion, or peri-tubular cuffs, are thin hollow cylinders (Fig. 1), and consist of collagen fibrils, hydroxypatite crystals, and water. The collagen molecules are long chains and they are arranged to form long triple helices called collagen fibrils. The hydroxypatite is a mineralized crystalline material. The bonding matrix or inter-tubular dentin also consists of collagen, mineral and water, but has different volume fractions than the peri-tubular dentin. The dentin composite considered here is, thus, assumed to be a two-phase, fibre-reinforced composite material. One phase is the peri-tubular inclusion and the other is bonding matrix.

The peri-tubular dentin (inclusion phase in Fig. 1) are built up of collagen fibrils and mineralized hydroxyapatite, in which a porous phase (liquid or gas) is embedded in a connected solid phase forming the skeleton. The solid phase is linear elastic and isotropic. The porous phase is saturated with a fluid at the pressure \( p_f \); or a gas at the pressure \( p_g \):

\[
\sigma = C \varepsilon + \sigma^g
\]

with

\[
C = \begin{cases} 
C_s & \text{in } \Omega^s, \\
0 & \text{otherwise,}
\end{cases}
\]

\[
\sigma^g = \begin{cases} 
0 & \text{in } \Omega^f, \\
-p^g + \chi(p^g - p_f) & \text{in } \Omega^g \cup \Omega^e,
\end{cases}
\]

where \( \varepsilon \) is microscopic strain, \( \chi \) is a parameter related to the degree of saturation and equals unity for fully saturated materials, \( C_s \) is the tensor of elastic moduli in the solid, \( \Omega^s \), \( \Omega^f \) and \( \Omega^e \) are domains occupied by the solid, fluid and gas phases, respectively. Their boundaries are denoted by \( \partial \Omega^s \), \( \partial \Omega^f \) and \( \partial \Omega^e \).

Fig. 1. Geometry of dentin composites and cell model: (a) Cross-section of composite cylinder assemblage; (b) unit cell model in axial direction; (c) cross-section of cell model.
The bonding matrix has similar structures as those of the inclusion phase but having different geometry and physical properties. Both of them are assumed to be isotropic and elastic.

Thus, both the inclusion and matrix phases, shown in Fig. 1c, are three-phase composites containing collagen fibrils, mineralized hydroxyapatite and liquid phase such as water or gas. Their effective properties need to be determined before we can predict the overall properties of the whole structure. To this end, consider an peri-tubular inclusion (or bonding matrix), which consists of collagen, mineralized hydroxyapatite and water. The effective properties of solid phase can be determined by the inclusion phase but having different geometry and physical properties. Both of them are assumed to be isotropic and elastic.

Having obtained the effective properties of peri-tubular dentin (or bonding matrix), the solid phase can now be viewed as a homogeneous porous material associated with inclusion phase, i.e., peri-tubular dentin. Bulk and shear moduli associated with inclusion phase, \( K_I \) and \( G_I \) are as well as moduli \( K_M \) and \( G_M \) for matrix phase can be calculated similarly.

Having obtained the effective properties of peri-tubular dentin (or bonding matrix), the solid phase can be used to predict the overall properties of porous composite material presented by Dormieux et al. [19] is used in our analysis. We briefly review this model and apply it to dentin composites below.

In the model, a representative volume element (RVE) \( \Omega \) is chosen so as to be statistically representative of the porous peri-tubular dentin, in particular, the characteristic size of heterogeneities is supposed to be small with respect to the dimension of RVE, which, in turn, is supposed to be small compared to the wavelength of the macroscopic structure.

In order to understand the key point of the homogenization procedure, let us consider an RVE consisting of the matrix material (solid phase) and porous phase. As an RVE is comprised of different materials, the micro-constitutive law that governs each material or phase in an RVE is given by Eq. (1). On the other hand, the macro-stress and macro-strain on the macro-level are directly associated with the global analysis of a porous material problem. On the macro-level, an RVE is just regarded as a point with a homogenized constitutive law. The macro-stress, \( \Sigma \), is usually defined as the volume average stress in an RVE, \( \langle \sigma \rangle \), as follows:

\[
\Sigma = \langle \sigma \rangle_O = \frac{1}{V_O} \int_O \sigma \, dV,
\]

where \( \Omega \) is the domain of the RVE and \( V_O \) is its volume. Similarly, the volume average strain in an RVE is defined by

\[
\varepsilon = \langle \varepsilon \rangle_O = \frac{1}{V_O} \int_O \varepsilon \, dV.
\]

However, as pointed out in [20], the above strain is equal to macro-strain, say \( \varepsilon \) (or \( E_I \) in the form of components), only if all phases of materials in an RVE are free of discontinuities. Using this definition, the following three types of boundary conditions are usually used to evaluate the overall material properties.

(a) Uniform traction on boundary \( \partial V \) of the RVE:

\[
\sigma_I n_j = \Sigma_I n_j.
\]

(b) Uniform displacement, \( U \), on boundary \( \partial V \) of the RVE. The component of a displacement vector in the RVE, \( u_i \), is given as

\[
u_i = E_I x_j.
\]

(c) Periodic conditions on boundary \( \partial V \) of the RVE:

\[
u_i = E_I x_j + u_i^p,
\]

where \( u_j \) are components of the outer normal unit vector, \( x_j \) are space coordinates, and \( u_i^p \) is periodic and represents the fluctuation part of the displacement.

After solving the boundary-value problem of an RVE under one of the above three boundary conditions, the micro-distribution of stress and strain within an RVE is determined, thus the remaining issue is to determine the strain/stress concentration tensor which is needed for calculating the effective material properties.

Keeping this in mind, we consider a porous material subjected to macroscopic strain \( \varepsilon \) and fluid pressure \( p^f \) (or gas pressure \( p^g \)). The boundary conditions applied to the porous material are

\[
\partial n = p^f \delta = -[p^g - \chi (p^g - p^f)] n \text{ on } \partial \Omega^f \cap \partial \Omega^g,
\]

\[
u_i = E_I x_j \text{ on } \partial \Omega^f,
\]
where $\delta$ is the unit tensor of second order. Since the material behaviour is linear, the superposition principle is used to decompose the load $(\mathbf{E}, \rho^e)\otimes (\mathbf{E}, \rho^e = 0)$ and $(\mathbf{E}, \rho^e = 0)$ [19]. Due to linearity, the local strain, $\varepsilon^e$, for the first loading case depends linearly upon the loading $\mathbf{E}$.

\[
\varepsilon^e(x) = A(x)\mathbf{E} \quad \text{in } \mathcal{O},
\]

(13)

where the superscript “$a$” stands for the quantity corresponding to the first loading case $(\mathbf{E}, \rho^e = 0)$, $A$ represents a concentration tensor to be determined. Using definition (6), the macroscopic stress is calculated as

\[
\langle \sigma^a \rangle = (1 - f) \langle \sigma^e \rangle_{\mathcal{O}}^a
= (1 - f) \langle C_e\varepsilon^e \rangle_{\mathcal{O}} = C_e \mathbf{E},
\]

(14)

where

\[
f = \frac{V_{\mathcal{O}}^e + V_{\mathcal{O}}^g}{V_0}, \quad C_e = (1 - f)C_a \langle A \rangle_{\mathcal{O}}^a
\]

(15)

with $C_e$ being effective stiffness moduli of the composite. Thus, the estimation of (14) and then $\langle A \rangle_{\mathcal{O}}^a$ is the key to predicting the effective moduli $C_e$. The approximation of $\langle A \rangle_{\mathcal{O}}^a$ through use of various micro-mechanics models will be discussed later.

For the loading case (b) $(\mathbf{E} = 0, \rho^e)$, the response should be proportional to $\rho^e$. Thus, a second-order tensor $\mathbf{B}$ exists such that

\[
\langle \sigma^b \rangle_{\mathcal{O}} = -\rho^e \mathbf{B}.
\]

(16)

The macroscopic stress $\Sigma = \langle \sigma \rangle_{\mathcal{O}}$ is obtained from (14) and (16), by superposition

\[
\Sigma = C_s \mathbf{E} - B \rho^e.
\]

(17)

Following the approach of [19], $\mathbf{B}$ can be determined as follows. Using Hill’s lemma [21,22] for the equilibrated stress field $\sigma^h$ and the strain field $\varepsilon^e$, we have

\[
\langle \sigma^h \varepsilon^e \rangle_{\mathcal{O}} = \langle \sigma^h \rangle_{\mathcal{O}} \langle \varepsilon^e \rangle_{\mathcal{O}}.
\]

(18)

By considering $\mathbf{E} = \langle \varepsilon^e \rangle_{\mathcal{O}}$, and $\sigma^h = C_e \varepsilon^e + \sigma^g$ one obtains

\[
-\rho^e \mathbf{B} \mathbf{E} = \langle \varepsilon^h \mathbf{C} \varepsilon^e \rangle_{\mathcal{O}} + \langle \sigma^g \rangle_{\mathcal{O}} \mathbf{E}.
\]

(19)

Since $\langle \varepsilon^h \rangle_{\mathcal{O}} = 0$, the first term of the right-hand side in (19) is equal to zero by Hill’s lemma. Thus, $\mathbf{B}$ can be written as

\[
\mathbf{B} = f \langle A \rangle_{\mathcal{O}} \mathbf{E} + (1 - f) \langle A \rangle_{\mathcal{O}} = I,
\]

(20)

From the identity

\[
\langle A \rangle_{\mathcal{O}} = (1 - f) \langle A \rangle_{\mathcal{O}} + f \langle A \rangle_{\mathcal{O}} = I,
\]

(21)

we have

\[
\mathbf{B} = \delta[I - (1 - f) \langle A \rangle_{\mathcal{O}}] = \delta[I - \mathbf{C}^{-1} \mathbf{C}_s].
\]

(22)

Therefore, the macroscopic constitutive law (17) is entirely defined once the macroscopic tensor of elastic moduli $\mathbf{C}_s$ has been determined.

In the case of an isotropic material, tensor $\mathbf{B}$ can be further written as

\[
\mathbf{B} = q_s \delta \quad \text{with } q_s = 1 - \frac{\lambda_s + \frac{2}{3} G_s}{\lambda_s + \frac{2}{3} G_s}
\]

(23)

and the macroscopic law (17) reads

\[
\begin{bmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{33} \\
\sigma_{12} \\
\sigma_{23} \\
\sigma_{31}
\end{bmatrix} =
\begin{bmatrix}
\lambda_s + 2G_s & \lambda_s & \lambda_s & 0 & 0 & 0 \\
\lambda_s & \lambda_s + 2G_s & \lambda_s & 0 & 0 & 0 \\
\lambda_s & \lambda_s & \lambda_s + 2G_s & 0 & 0 & 0 \\
0 & 0 & 0 & 2G_s & 0 & 0 \\
0 & 0 & 0 & 0 & 2G_s & 0 \\
0 & 0 & 0 & 0 & 0 & 2G_s
\end{bmatrix},
\]

(24)

where $\lambda_s$ and $G_s$ are two Lamé constants given by

\[
\lambda_s = \frac{E_s \nu_s}{(1 + \nu_s)(1 - 2\nu_s)}, \quad G_s = \frac{E_s}{2(1 + \nu_s)}
\]

(25)

with $E_s$ and $\nu_s$ being effective Young’s modulus and Poisson’s ratio.

In addition, the variation of porosity with stress and pore pressure is given by

\[
f - f_0 = f \delta \langle \varepsilon \rangle_{\mathcal{O}} \otimes \rho^e = q_s \text{tr}(\mathbf{E}) + \frac{(q_s - f) \rho^e}{\lambda_s + \frac{2}{3} G_s},
\]

(26)

where $f_0$ is the initial porosity before loading.

3. Calculation of concentration tensor $A$

In most situations, the porous phase can be considered as (hollow) inclusions embedded in the solid phase. As such, it is assumed that the pores are connected by narrow channels which have a negligible effect on the overall behaviour. Therefore, a reasonable estimate of the effective moduli can be made by considering the porous phase as inclusions distributed in the solid phase either randomly or periodically. Consequently, the porous material is completely characterized by the volume fraction of the pores, the pore shape and the orientation. The presence of pores affects
the overall mechanical and physical properties of the material. In order to calculate the effective moduli of a porous material, the material can be treated as a composite medium composed of a homogeneous matrix and a large number of ellipsoidal inclusions. Following the standard micro-mechanics approach [23], the following relation exists between the globally applied strain \( \varepsilon \) and the local strain in the solid phase \( \varepsilon' \):

\[ \varepsilon'(x) = A'(x) \varepsilon, \quad (27) \]

where \( A'(x) \) is the strain concentration tensor for the solid phase. The concentration tensor \( A \) has the following properties:

\[ \langle \varepsilon \rangle_A = f \langle A^{f+g} \rangle_A + (1-f) \langle A \rangle_A = \varepsilon. \quad (28) \]

It follows that

\[ f \langle A^{f+g} \rangle_A + (1-f) \langle A \rangle_A = \mathbf{I}. \quad (29) \]

The tensor \( A' \) can be determined from (29). The pore strain concentration tensor \( A^{f+g} \) is evaluated using the Mori–Tanaka approach (see [24,25] for details)

\[ A^{f+g} = [I - (1-f)S]^{-1} \quad (30) \]

where \( S \) is the Eshelby tensor [26] some examples of which have been given in Appendix A.

4. Micro-mechanical model of dentin composites

In what follows, the dilute micro-mechanics model presented in [11] is extended such as to be able to capture the mechanical properties of the dentin materials and analyse effects of porous phase on the effective properties of the dentin materials. In the dilute applications, we assume that the interaction among inclusions in an infinite domain can be ignored. While the interaction can be taken into account by using more complex micro-mechanics model such as a generalized self-consistent inclusion-matrix-composite model which is underway and will be given later. As shown in Fig. 1, the model used in our analysis consists of a circular cylinder of liquid or gas phase surrounded by two layers of solid phase (the inner one is the inclusion phase and the outer layer is the matrix phase) in the form of similar cylindrical shell.

Following the way described in [11], consider a fibre-reinforced porous composite as shown in Fig. 1. For random fibre arrangement, the general elastic feature of the composite is assumed to be transversely isotropic. The strain–stress equation for a transversely isotropic porous material can be written in terms of six material constants in the form

\[
\begin{bmatrix}
\sigma_{11} \\
\sigma_{22} \\
\sigma_{33} \\
\sigma_{12} \\
\sigma_{23} \\
\sigma_{31}
\end{bmatrix} =
\begin{bmatrix}
C_{11} & C_{12}^{*} & C_{13}^{*} \\
C_{12}^{*} & C_{22}^{*} & C_{23}^{*} \\
C_{13}^{*} & C_{23}^{*} & C_{33}^{*}
\end{bmatrix}
\begin{bmatrix}
\varepsilon_{11} \\
\varepsilon_{22} \\
\varepsilon_{33} \\
\gamma_{12} \\
\gamma_{23} \\
\gamma_{31}
\end{bmatrix} +
\begin{bmatrix}
1 \\
1 \\
1 \\
1 \\
1 \\
1
\end{bmatrix}
\begin{bmatrix}
q^{f} p^{e} \\
q^{f} p^{e} \\
q^{f} p^{e} \\
q^{f} p^{e} \\
q^{f} p^{e} \\
q^{f} p^{e}
\end{bmatrix},
\]

As was treated in [11], we use the following six constants in our analysis:

\[ K_{23} = \frac{1}{2} (C_{22}^{*} - C_{23}^{*}), \]

\[ G_{23} = \frac{1}{2} (C_{22}^{*} - C_{23}^{*}), \]

\[ G_{12}^{*} = G_{13}^{*} = G_{1}^{*} = C_{44}^{*}, \]

\[ E_{1}^{*} = C_{11}^{*} - \frac{2(C_{12}^{*})^2}{C_{22}^{*} + C_{23}^{*}} \]

\[ C_{11}^{*}, \text{ and } q^{f}. \] Here \( K_{23}^{*}, \text{ and } G_{23}^{*}\) are a bulk and shear modulus, respectively, in the \( x_2-x_3 \)-plane; \( G_{1}^{*} \) is a shear modulus governing shear in any plane normal to the \( x_2-x_3 \)-plane; \( E_{1}^{*} \) is the longitudinal Young’s modulus, and \( C_{11}^{*} \) is associated with axial stress or strain in the \( x_1 \)-direction, while lateral deformation is prevented by a rigid enclosure. As was treated in Section 2, the superposition principle is used to decompose the load \( (E_{0} p^{0} \text{ in the form of components}) \) into two elementary loadings \( (E_{0} p^{0} = 0) \) and \( (E = 0, p^{0}) \) [19]. For a dilute distribution of inclusion phase and \( p^{0} = 0 \), Hashin and Rosen [11] have given the first five material constants as below:

\[ K_{23} = K_{M} \frac{\phi(1-x^{2})(1+2\nu_{1}f_{1}) + 2\nu_{1}f_{1}(1+x^{2}/2\nu_{1})}{\phi(1-x^{2})f_{1} + (f_{1}+2\nu_{1})(1+x^{2}/2\nu_{1})}, \]

\[ G_{1}^{*} = G_{M} \frac{\eta(1-x^{2})(1+f_{1}) + f_{M}(1+x^{2})}{\eta(1-x^{2})f_{1} + (1+x^{2})(1+f_{1})}. \]

\[ E_{1}^{*} = f_{1}E_{1} + f_{M}E_{M}, \]

\[ C_{11}^{*} = E_{1}^{*} + 4\nu_{1}^{2}K_{23}^{*}, \]

\[ G_{23}^{(o)} = G_{M} \left( 1 - \frac{2(1-\nu_{1})}{1-2\nu_{1}} f_{1} A_{2}^{*} \right), \]

\[ G_{23}^{(l)} = G_{M} \left( 1 + \frac{2(1-\nu_{1})}{1-2\nu_{1}} f_{1} A_{2}^{*} \right), \]
where \( v_M \) and \( v_1 \) are Poisson’s ratios of matrix and inclusion phases, the superscripts “(u)” and “(l)” stand for upper and lower bounds of a variable, \( A_u^j \) and \( A_l^j \) are determined from equations given in Appendix B, and

\[
\hat{K}_r = \lambda_r + G_r \gamma = I, M),
\]

\[
\phi = \frac{K_1}{K_M} = a, \quad f_1 = \frac{b^2}{c^2},
\]

\[
f_M = 1 - f_1, \quad \eta = \frac{G_1}{G_M},
\]

\[
f_1 = \frac{b^2 - a^2}{c^2}, \quad v_1^* = \frac{f_1 E_1 L_1 + f_M E_M L_2 v_M}{f_1 E_1 L_3 + f_M E_M L_2},
\]

\[
L_1 = 2 f_1 (1 - v_M) \eta + f_M (1 + v_M) \gamma_M,
\]

\[
L_2 = f_1 [(1 + v_1) x^2 + 1 - v_1 - 2 \gamma_1],
\]

\[
L_3 = 2 (1 - v_M) \eta + f_M (1 + v_M).
\]

The first five material constants in Eqs. (32)–(35) can, thus, be determined using the formulation described above. From these five material constants any desired elastic constant can be obtained. Important derived constants are

\[
E_2^* = E_3^* = \frac{4G_{23}K_{23}}{K_{23}^* + \psi G_{23}^*},
\]

\[
v_2^* = \frac{K_{23} - \psi G_{23}}{K_{23}^* + \psi G_{23}^*},
\]

with

\[
\psi = 1 + \frac{4K_{23}^*(v_1^*)^2}{E_1^*},
\]

where \( E_2^* \) (or \( E_3^* \)) is the transverse Young’s modulus in the plane perpendicular to the axial direction, and \( v_{23}^* \) is the transverse Poisson’s ratio in the same plane.

For the loading case \( E = 0 \) and \( p^* \), the determination of \( q^* \) is similar to that in Section 2, we omit those details here. The final result for the effective pore concentration coefficient \( q^* \) is given by

\[
q^* = \frac{1}{2} \left( f_1 q_1 + (1 - f_1) q_M + \frac{q_1 q_M}{f_1 q_M + (1 - f_1) q_1} \right).
\]

5. Numerical results and discussion

The approach presented here is to initially predict the elastic properties of mineral in the form of carbonated apatite which is the major component of the peri- and the inter-tubular materials using those results with measurements obtained by Kinney et al. [13] and Marshall et al. [27] using atomic force microscopy-based nano-indentation equipment. The mineral phase of dentin is considered to contain typically 5% of carbonate and to have a somewhat disordered crystalline form, more so than enamel [28]. As such, the intrinsic elastic properties of this phase are anticipated to be lower than that of fully dense hydroxyapatite but are at present unknown. In this case, there are two approaches to this problem, we could assume values of \( E, v \) for collagen and knowing the effective values and the volume fractions of these phases and porosity use Eqs. (15) and (23) above to estimate the modulus and Poisson’s ratio of the HAP, or assume values of \( E, v \) for both collagen and HAP to determine the modulus and Poisson’s ratio of the composite. Following this, the spatial variation of the properties of dentin from the pulp to the dentin–enamel junction (DEJ) are presented.

Since neither information concerning the relative amounts of collagen and carbonated apatite, nor values of \( E \) and \( v \) for collagen and carbonated apatite of the peri- and the inter-tubular dentin was reported in the literature, which are required by the present model, it is necessary to choose specific values. For inter-tubular dentin, the values \( E_{\text{peri}} = 1.2 \) GPa and \( v_{\text{peri}} = 0.35 \) for collagen were chosen, based on the values reported for bone materials whose elastic behaviour is roughly comparable to collagen in dentin [29]. The area fractional values recommended in [30] were used in our analysis, i.e., the inter-tubular dentin composition was \( A_e = 0.3 \) collagen, \( A_{\text{apatite–peri}} = 0.5 \) mineral, and \( f_{\text{pore}} = 0.28 \) porosity. In addition, the value \( v_{\text{apatite–peri}} = 0.28 \) was also assumed in order to use the proposed model.

Using Eqs. (15) and (23), with \( E_{\text{peri}} = 18.3 \) GPa as obtained by atomic force microscopy [27] and the values described above, the values of \( E_{\text{apatite–peri}} \) for carbonated apatite and \( v_{\text{peri}} \) for inter-tubular dentin are obtained as

\[
E_{\text{apatite–peri}} = 35.83 \, \text{GPa}, \quad v_{\text{peri}} = 0.24, \quad q_{\text{peri}} = 0.44,
\]

where \( E_{\text{peri}}, v_{\text{peri}} \) and \( q_{\text{peri}} \) are the related material properties of inter-tubular dentin. In the calculation, it is found that the variation of \( E_{\text{apatite–peri}} \) has no effect on the values of \( v_{\text{peri}} \) and \( q_{\text{peri}} \). If the same values of the carbonated apatite and collagen are assumed for the peri-tubular dentin, the area fraction of collagen can be determined using the present model. Please note that this phase usually consists of approximately 5% porosity and nearly 90% mineral. With the value of \( E_{\text{peri}} = 22.5 \) GPa [13], Eqs. (15) and (23) indicate the following:

\[
A_e = 0.33, \quad v_{\text{peri}} = 0.288 \quad \text{and} \quad q_{\text{peri}} = 0.138,
\]

in which the porosity \( f_{\text{pore}} = 0.05 \) is assumed. It is noted from the data given in [31] that \( x^2 = a^2/b^2 \) is independent of position although both \( a \) and \( b \) vary from the pulp to the DEJ. Thus, \( E_{\text{peri}} \) does not vary with the position, as it is a function of \( x^2 \) only when the tubular diameter changes with its location. This conclusion is consistent with that reported from Kinney et al. [13].
Alternatively, using the values listed above, i.e., $E_c = 1.2\text{ GPa}$, $n_c = 0.3$, $E_{\text{apatite}} = 35.83\text{ GPa}$, and $n_{\text{apatite}} = 0.28$, then the porosity and collagen dependence of the peri-tubular cuff or inter-tubular dentin is shown in Figs. 2 and 3. Typically Fig. 2 illustrates the effects of porosity on Effective Young’s modulus, Poisson’s ratio, and pore concentration coefficient $q_a$, respectively. It can be seen from Fig. 2 that both the Young’s modulus and Poisson’s ratio decrease along with the increase in porosity, but the pore concentration coefficient $q_a$ increases with the increase in porosity. Fig. 3 shows the variation of the three effective constants above with the volume fraction of collagen when the porosity is equal to 0.05. It is found from Fig. 3 that both the Poisson’s ratio and pore concentration coefficient $q_a$ increase slowly when the volume fraction of collagen increases, while the Young’s modulus decreases along with the increase in the volume fraction of collagen, as expected.

Having analysed the mechanical behaviour for both peri- and inter-tubular dentin, we are now in a position to predict the elastic properties of the composite dentin material. It should be mentioned that Pashley [31] reported the dimensions of the lumen, peri-tubular cuff as well as the density of tubules at different locations through the tooth. These are listed in Table 1 for completeness. With the values of $E$ for the peri- and inter-tubular dentin recorded in [13,27] and the dimensions of these components the elastic properties are determined from the micro-mechanics model in Section 4, which is shown in Figs. 4–6. In the calculation, since the material constants required in the proposed formulation are not available in the literature, the dentin composite which has been analysed above is used whose material constants are:

1) **Peri-tubular dentin**: Young’s modulus $E_{c-\text{peri}} = 1.2\text{ GPa}$, Poisson’s ratio $n_{c-\text{peri}} = 0.3$, $f_{\text{poro}} = 0.05$, $E_{\text{apatite-peri}} = 66.76\text{ GPa}$, $n_{\text{apatite-peri}} = 0.27$, and volume of collagen = 0.1.

2) **Inter-tubular dentin**: Young’s modulus $E_{c-\text{int}} = 0.94\text{ GPa}$, Poisson’s ratio $n_{c-\text{int}} = 0.30$, and $f_{\text{poro}} = 0.1$, $E_{\text{apatite-int}} = 35.8\text{ GPa}$, $n_{\text{apatite-int}} = 0.25$, and volume of collagen = 0.3.

### Table 1

<table>
<thead>
<tr>
<th>Distance from pulp (mm)</th>
<th>$A_i$</th>
<th>$A_p$</th>
<th>$A_i$</th>
<th>$E_{a-\text{peri}}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1–0.5</td>
<td>12.19</td>
<td>36.58</td>
<td>51.23</td>
<td>22.5</td>
</tr>
<tr>
<td>0.6–1.0</td>
<td>7.64</td>
<td>22.92</td>
<td>69.44</td>
<td>22.5</td>
</tr>
<tr>
<td>1.1–1.5</td>
<td>3.96</td>
<td>11.39</td>
<td>84.15</td>
<td>22.5</td>
</tr>
<tr>
<td>1.6–2.0</td>
<td>2.85</td>
<td>8.55</td>
<td>88.60</td>
<td>22.5</td>
</tr>
<tr>
<td>2.1–2.5</td>
<td>1.46</td>
<td>4.39</td>
<td>94.15</td>
<td>22.5</td>
</tr>
<tr>
<td>2.6–3.0</td>
<td>1.01</td>
<td>3.01</td>
<td>95.98</td>
<td>22.5</td>
</tr>
<tr>
<td>3.1–3.5</td>
<td>0.96</td>
<td>2.86</td>
<td>96.18</td>
<td>22.5</td>
</tr>
</tbody>
</table>

aData based on values from [32] as tabulated in [31], and $E_{a-\text{peri}}$ is from [13].
Using Eqs. (36)–(43), the elastic properties of the dentin composite are estimated.

Fig. 4 show effective material constants $K_{23}^*$, $G_1^*$, $E_1^*$ and $q^*$ as functions of the volume fraction of peri-tubular dentin for the dentin composite described above (i.e., the effects of the thickness of peri-tubular dentin on the overall material property). As was expected, all material constants, except for pore concentration coefficient $q^*$, increase for an increase in the volume fraction of peritubular dentin. The four effective properties above are also shown in Fig. 5 as a function of the distance from the pulp. It is observed from Fig. 5 that the material properties will decrease quickly at the beginning and then keep nearly constant when the distance is greater than 1.3 mm. The difference between modulus $E_1^*$ (parallel) and $E_2^*$ (perpendicular) is shown in Fig. 6. It is found from the figure that both of them decrease along with an increase in the distance from the pulp. It is also observed that $E_1^*$ is always greater than $E_2^*$.

In order to allow for comparisons with Kinney’s results [13], The results listed in Table 2 are obtained using the dentin composite which has been analysed by nano-indentation method [13,27] whose material constants are:

1. Peri-tubular dentin: Young’s modulus $E_{2\text{-peri}} = 22.5$ GPa, Poisson’s ratio $\nu_{2\text{-peri}} = 0.25$, and
2. Inter-tubular dentin: Young’s modulus $E_{2\text{-int}} = 15$ GPa, Poisson’s ratio $\nu_{2\text{-int}} = 0.1$, while $q_{2\text{-peri}} = 0.135$ and $q_{2\text{-int}} = 0.44$ is assumed. Some typical results of $E_1^*$ are listed in Table 2 and comparison is made with those predicted by the formulation in [13]. It is apparent that they are in good agreement.

6. Conclusions

Micro-mechanics theory can be a powerful tool to estimate the effective material properties and mechanical behaviour of dentin composites in terms of its constituents, and can also provide important information concerning the micro-organization and composition of the dentin, a natural composite.

A micro-mechanics cell model has been developed for estimating effective material properties of dentin composites which may be composed of a circular hollow cylinder filled with liquid or gas phase, which is surrounded by two circular cylindrical shells, a thin shell and a matrix phase. Each layer of cylindrical shell is here considered as a composite consisting of collagen fibrils, with mineralized hydroxyapatite, loosely connected to their neighbours, and water (or gas in the case of dry dentin composite). The cell model was developed based on the dilute micro-mechanics model [11] and is able to capture the mechanical properties of natural dentin and analyse the effects of mineral content, porosity, thickness of peri-tubular layer, and porous phase on the effective material properties of dentin composites. Numerical results are presented to compare predictions obtained by the proposed approach and the by the approach in [13]. This study also shows that knowing the volume fraction of the various components of the peri-tubular and inter-tubular materials as well as their $E$ modulus values, the proposed formulation can estimate a self consistent value of the $E$ (or other) modulus of either the apatite or the collagen from one of their $E$ (or other) values.

<table>
<thead>
<tr>
<th>Tubular concentration</th>
<th>Present results</th>
<th>Results of Ref. [13]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>15.37</td>
<td>15.34</td>
</tr>
<tr>
<td>0.10</td>
<td>15.76</td>
<td>15.80</td>
</tr>
<tr>
<td>0.15</td>
<td>16.13</td>
<td>16.19</td>
</tr>
<tr>
<td>0.2</td>
<td>16.50</td>
<td>16.57</td>
</tr>
</tbody>
</table>
Acknowledgements

This work was financially supported by the Australian Research Council and partially by a National Health and Medical Research Council Oral Initiative grant. These supports are gratefully acknowledged.

Appendix A. Eshelby’s tensor for ellipsoidal and spherical inclusions [26]

\[ S_{111} = \frac{3}{8\pi(1-v)} a_1^2 F_{11} + \frac{1-2v}{8\pi(1-v)} F_1, \]  
\[ S_{112} = \frac{1}{8\pi(1-v)} a_2^2 F_{12} - \frac{1-2v}{8\pi(1-v)} F_1, \]  
\[ S_{113} = \frac{1}{8\pi(1-v)} a_3^2 F_{13} - \frac{1-2v}{8\pi(1-v)} F_1, \]  
\[ S_{121} = \frac{1}{16\pi(1-v)} (a_1^2 + a_2^2) F_{12} + \frac{1-2v}{16\pi(1-v)} (F_1 + F_2), \]
\[ S_{ikm} = S_{jkm} = S_{ymk}, \]

where

\[ F_1 = \frac{4\pi a_1 a_2 a_3}{(a_1^2 - a_2^2)(a_1^2 - a_3^2)^{1/2}} [E(\theta, k) - G(\theta, k)], \]
\[ F_3 = \frac{4\pi a_1 a_2 a_3}{(a_2^2 - a_3^2)(a_1^2 - a_3^2)^{1/2}} \times \left[ \frac{a_2(a_1^2 - a_3^2)^{1/2}}{a_1 a_3} - G(\theta, k) \right], \]
\[ F_1 + F_2 + F_3 = 4\pi, \quad 3F_{11} + F_{12} + F_{13} = \frac{4\pi}{a_1^2}, \]
\[ 3a_1^2 F_{11} + a_2^2 F_{12} + a_3^2 F_{13} = 3F_1, \]
\[ F_{12} = (F_2 - F_1)/(a_1^2 - a_2^2), \]

with

\[ E(\theta, k) = \int_0^\theta \frac{dw}{(1 - k^2 \sin^2 w)^{1/2}}, \]
\[ G(\theta, k) = \int_0^\theta \frac{dw}{(1 - k^2 \sin^2 w)} \mathrm{d}w, \]
\[ \theta = \sin^{-1}(1 - a_3^2/a_1^2)^{1/2}, \]
\[ k = [(a_1^2 - a_2^2)/(a_1^2 - a_3^2)]^{1/2} \]

and all other non-zero components are obtained by the cyclic permutation of (1, 2, 3). The components which cannot be obtained by the cyclic permutation are zero, for instance, \( S_{112} = S_{223} = S_{313} = 0 \).

\[ A.2. \text{ Sphere (a_1 = a_2 = a_3)} \]
\[ S_{111} = S_{222} = S_{333} = \frac{7 - 5v}{15(1-v)}, \]  
\[ S_{122} = S_{223} = S_{331} = S_{113} = \frac{5v - 1}{15(1-v)}, \]  
\[ S_{121} = S_{232} = S_{313} = \frac{4 - 5v}{15(1-v)}. \]

Appendix B. Equations for \( A_4^1 \) and \( A_5^4 \) in Eqs. (40) and (41) [11]

B.1. Equations for calculating \( A_4^1 \)
\[ A_1^1 + f_1^{-1} A_2^1 + f_1^2 A_3^1 + f_1 A_4^1 = 1, \]
\[ \frac{3 - 4v}{3 - 2v} f_1^{-1} A_2^1 - 2f_1^2 A_3^1 + \frac{f_1}{1 - 2v} A_4^1 = 0, \]
\[ A_1^1 + A_2^1 + A_3^1 + A_4^1 - B_1^1 - B_2^1 - B_3^1 - B_4^1 = 0, \]
\[ \frac{3 - 4v}{3 - 2v} A_2^1 - 2A_1^1 + \frac{1}{1 - 2v} A_3^1 + \frac{3 - 4v}{3 - 2v} B_2^1 + 2B_3^1 - \frac{1}{1 - 2v} B_4^1 = 0, \]
\[ A_1^1 + A_2^1 - 3A_3^1 + \frac{1}{1 - 2v} A_4^1 - \eta B_1^1 - 3\eta B_3^1 + 3\eta B_4^1 - \frac{\eta}{1 - 2v} B_4^1 = 0, \]
\[ \frac{1}{3 - 2v} A_2^1 + 2A_3^1 - \frac{1}{1 - 2v} A_4^1 + \frac{\eta}{3 - 2v} B_2^1 - 2\eta B_3^1 + \frac{\eta}{1 - 2v} B_4^1 = 0, \]
\[ B_1^1 + \frac{3\alpha_1^2}{3 - 2v} B_2^1 - 3\alpha_1^4 B_3^1 + \frac{\alpha_1^{-2}}{1 - 2v} B_4^1 = 0, \]
\[ \frac{\alpha_1^2}{3 - 2v} B_2^1 + 2\alpha_1^4 B_3^1 - \frac{\alpha_1^{-2}}{1 - 2v} B_4^1 = 0. \]

B.2. Equations for calculating \( A_5^4 \)
\[ A_1^4 + \frac{3f_1^{-1}}{3 - 2v} A_2^4 - 3f_1^2 A_3^4 + \frac{f_1}{1 - 2v} A_4^4 = 1, \]
\[ \frac{f_1^{-1}}{3 - 2v} A_2^4 + 2f_1^2 A_3^4 - \frac{f_1}{1 - 2v} A_4^4 = 0. \]
The remaining six equations have the same form as those of Eqs. (B.3)–(B.8) except that the superscript “\(e\)” is replaced by “\(s\)”.

References