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Effects of aging on the mechanical behavior of human dentin ☆

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Abstract

An experimental study on the mechanical behavior of human dentin and the influence of age was conducted. Beams with rectangular cross-section were sectioned from the coronal dentin of virgin extracted molars ($N=76$) that were obtained from ($N=70$) patients between 17 and 80 years of age. The beams were loaded in either quasi-static 4-point flexure or 4-point flexural fatigue to failure and the stiffness, strength and fatigue properties were evaluated. In characterizing the fatigue response the beams were divided into two age groups that were regarded as young ($17 \leqslant \text{age} \leqslant 30$, mean ± std. dev. = 25 ± 5 years) and old ($50 \leqslant \text{age} \leqslant 80$, mean ± std. dev. = 64 ± 9 years) dentin. Results from monotonic loading showed that both the flexural strength and strain to fracture of dentin decreased significantly with age. The fatigue life of dentin increased with a reduction in cyclic stress amplitude and the fatigue strength of young dentin was greater than that of old dentin at all cyclic stress amplitudes. The endurance strength of young dentin (at 10^7 cycles) was approximately 44 MPa, whereas the old dentin exhibited an endurance strength of approximately 23 MPa.Based on differences in the mechanical behavior and microscopic features of the fracture surfaces from the young and old specimens, aging appears to result in an increase in both the rate of damage initiation and propagation in dentin.

Keywords: Age; Dentin; Fatigue; Fracture

1. Introduction

With improvements in preventative dentistry and the decline of caries in the young, the number of fully dentate patients is increasing [1,2]. But despite many optimistic reports, dental caries and tooth fracture remain a problem in older adults [3,4]. The occurrence of crown and root caries is as high as 65% in patients exceeding 65 years of age [3,5,6]. In general, seniors have a larger number of restored teeth and they are at a greater risk of recurrent decay or fracture [6].

There are natural changes that occur to human teeth with aging. Perhaps most notable are the changes in physical and chemical structure of dentin [7]. Dentin is a hard tissue that comprises the bulk of the tooth and is approximately 45% inorganic, 35% organic, and 20% water by volume [7]. On a microscopic scale dentin is comprised of intertubular dentin, and the highly mineralized cylindrical cuffs of peritubular dentin surrounding the dentin tubules [7]. The tubules exist as open channels with 1–2 μm internal diameter and extend radially from the pulp throughout the dentin. There is a continual increase in thickness of the peritubular dentin from approximately 10 weeks in utero to old age that results from deposition of mineral within the lumen [8]. The tubule lumens decrease in diameter and can undergo complete occlusion [7,9]. According to Webber [10], up to 50 percent of the dentinal tubules can become completely occluded with age under natural physiological conditions. With the reduction in lumen diameter there is a decrease in permeability of dentin [11]. In fact, Toto et al. [12] found that teeth over 50 years contained less water than young teeth (10–20 years of age). Dehydrated dentin has a lower toughness and has been

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described as being brittle [13–15]. Based on the recognized evolution in structure of dentin with age, changes in the mechanical behavior are not unlikely.

The majority of studies on the mechanical behavior of dentin have focused on the strength and stiffness (e.g. [16,17]), which are most relevant to acute overloads to failure [18]. Kinney et al. [19] recently reviewed the mechanical properties of dentin, which revealed that damage accumulation, crack growth and fracture are likely contributors to tooth fracture. Very few studies have been reported in this area. Tonami and Takahashi [20] evaluated the fatigue properties of dentin and determined that the endurance strength of bovine dentin from young (2–3 years) and adult (3.5–6 years) animals was 46.9 and 51.0 MPa, respectively. More recently, Nalla et al. [21] reported that the fatigue strength of human dentin increased with decreasing stress amplitude and that dentin exhibits an endurance limit that ranges between 25 and 45 MPa. An evaluation of the results with respect to frequency, time and load cycles implied that the fatigue behavior of dentin was both cyclic and time dependent. Nalla et al. [22] also showed that the fatigue strength of human dentin decreased with an increase in mean stress. Though the aforementioned studies have provided new knowledge on the fatigue properties of dentin, the influence of age has not been addressed. In this investigation the mechanical behavior of dentin was evaluated in quasi-static and cyclic flexure. The primary objective of the study was to determine if the fatigue strength of human dentin is dependent on age.

2. Materials and methods

Human second and third molars were obtained from participating clinics in the state of Maryland according to protocols approved by the University of Maryland Institutional Review Board. The patient's sex and age were recorded and the teeth were stored in Hanks Balanced Salt Solution (HBSS) at 2 °C immediately after extraction. An inspection for visible decay or structural defects was conducted at extraction and throughout the sectioning process. The teeth were cast in a polyester resin foundation and uniform sections of 1.5 mm thickness were obtained along the mesial–distal or bucccal–lingual axis using a numerically controlled slicer/grinder[1] with continuous flood coolant. Primary slices were obtained equidistant from the mesial–distal or buccal–lingual aspect using diamond impregnated slicing wheels (#320 mesh abrasives). Each of the sections was mounted to a glass sheet with temporary adhesive[2] for secondary sectioning. Beams of rectangular cross-section (0.5 × 1.5 mm) and 8 mm length were obtained from the coronal dentin such that the dentin tubules were oriented perpendicular to the longitudinal axis (Fig. 1). After machining, the beams were stored in HBSS at 22 °C for an average of 4 days, with an overall range between 1 and 12 days.

The dentin specimens were loaded in four-point flexure to failure using ether monotonic or fatigue loads. In evaluating the flexure properties of dentin under monotonic loads, a total of 37 dentin beams were obtained from 17 molars of 15 different patients; typically the properties of each specific age were obtained from the molars of a single person. According to results from monotonic loading the teeth were divided into groups of young ($17 \leqslant \text{age} \leqslant 30$, mean ± std. dev. = 25 ± 5 years) and old ($50 \leqslant \text{age}$, mean ± std. dev. = 64 ± 9 years) dentin. A total of 59 human molars from 55 patients were sectioned into 88 fatigue specimens; 50 of the specimens ranged from 17 to 30 years in age and 38 specimens were from patients 50 years or older. In general, either 1 or 2 beams were obtained from each tooth while no more than 3 beams were obtained from any single molar. While some of the old dentin specimens were transparent (sclerotic) others were not, and the degree of transparency did not appear constant among similar aged teeth. Thus, patient age appeared to provide an objective measure of aging rather than transparency. To the author's knowledge no previous studies have been reported on the influence of age to the mechanical behavior of human dentin. Thus,

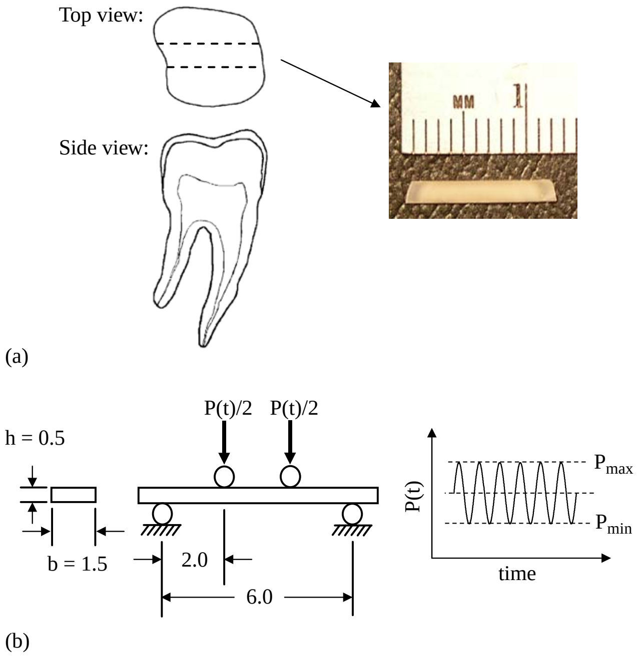

Fig. 1. Schematic diagram illustrating the location at which dentin beams were sectioned and the corresponding load configuration. (a) sectioning of a dentin beam. (b) beam geometry and load configuration (dimensions in mm).

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[1] Model S3818EL, K.O.LEE Company, Aberdeen, SD.

[2] Crystalbond™ 509, Aremco Products, Inc.,Valley Cottage, NY.

the two age groups were used as a simple means to categorize the teeth and were not based on any expected changes in properties pertaining to a specific age.

All experiments were conducted using a universal testing system[3], which has a load capacity and sensitivity of 225 and ±0.01 N, respectively. The specimen test geometry and flexure apparatus conformed to a scaled version of ASTM D790M for four-point flexural testing of materials with $\frac{1}{3}$ load span arrangement [23]. All experiments were conducted with the specimens fully immersed in a HBSS bath (22 °C) to maintain hydration. Quasi-static flexure was performed using displacement control loads to failure with a cross-head rate of 0.001 mm/s. The rate was established from preliminary experiments and resulted in a reasonable test duration for the range in bend displacements required for failure. The load and load-line displacement history were monitored throughout the flexure experiments using the EnduraTEC control software at a frequency of 0.25 Hz (1 data point every 4 s). The load at failure and beam geometry were used to estimate the bend stress according to conventional beam theory. Similarly, the bend strain in the dentin beams was estimated in terms of the four-point flexure arrangement and load-line displacement assuming a constant radius of curvature. The flexural response of the dentin beams under quasi-static loading to failure was examined in terms of stress–strain diagrams and the flexure strength and energy to fracture were determined from the maximum bend stress and area under the stress–strain diagrams, respectively.

In examining the fatigue properties of dentin, the beams were subject to load control actuation at a frequency of 5 Hz using a stress ratio (R) of 0.1. A loading frequency of 5 Hz was chosen to balance concerns associated with the frequency of mastication and the duration of time required to complete the individual tests. Fatigue loads were selected to achieve a maximum cyclic stress between 30% and 95% of the flexure strength for that age group. The experimental evaluation was initiated using large cyclic stresses (e.g., 95% of the average flexural strength) and successive specimens were tested with stress amplitude decreased by 5–10% until reaching a stress amplitude that did not cause fracture within 10^6–10^7 cycles. Beams of young dentin were subjected to maximum cyclic stresses ranging between 90 and 165 MPa. The old dentin was examined under stress amplitudes ranging from 50 to 140 MPa. Both the bend load and load-line displacement were monitored at 150 Hz for 0.2 s (1 cycle) at specific increments of fatigue loading over the entire fatigue life. Measurements were generally obtained between every 5 and 25 k cycles. The stress-life response for the dentin specimens within each age group was developed by plotting the cumulative results for all beams in terms of the cyclic stress amplitude and number of cycles to failure. Note that specimens obtained from the same tooth were not examined at the same cyclic stress level and no more than three specimens were obtained from a single tooth.

The fatigue life distribution of dentin was modeled according to a power law in the form

$$\sigma = A(N)^B \tag{1}$$

where A and B are the fatigue life coefficient and exponent, respectively, σ is the stress amplitude and N is the number of cycles to failure. Similar to the characterization of conventional engineering materials, an endurance limit (also regarded as the "fatigue limit") was estimated for both young and old dentin by the cyclic stress amplitude at which the number of cycles to failure reached a horizontal asymptote. If not directly evident from the fatigue life diagram (through evidence of a horizontal asymptote) the equivalent endurance strength was estimated from the power law model at 10^7 cycles.

The bend stress imposed by fatigue loading was obtained directly from the flexure formula. Changes in the flexural modulus and stiffness of the dentin beams that resulted from fatigue loading were analyzed using the load and load-line displacement history. The stiffness (S) of the dentin beams was estimated using a linear trend line fit to data retrieved over the entire load cycle and the flexural modulus (E) was estimated directly from the bend stiffness. For the four-point flexure arrangement with $\frac{1}{3}$ load span configuration, the flexural modulus can be estimated according to

$$E = \frac{0.21L^3S}{bh^3}, \tag{2}$$

where the quantities L, S, b, and h represent the length between the external beam supports, the slope obtained from the load versus the load-line displacement history, and the beam width and beam height, respectively (Fig. 1b). The dentin beams were observed to undergo changes in E with fatigue loading that warranted a quantitative description. For many advanced engineering materials a change in elastic modulus with cyclic loading is used to characterize the initiation and propagation of damage. Therefore, an evaluation of E over the fatigue life provided a means for identifying differences in the fatigue process between the young and old dentin.

Following failure, each specimen was inspected using a scanning electron microscope[4] (SEM) to confirm the tubule orientation and to examine the fracture surface. The beams were allowed to dehydrate in air, sputtered with gold palladium to enhance conductance and

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[3] EnduraTEC Model ELF 3200, Minnetonka, MN.

[4] JEOL Model JSM-5600, Peabody, Massachusetts.

evaluated in secondary electron imaging (SEI) mode with an accelerating voltage of 20 KV. Both the tensile and compressive sides, as well as the fracture surfaces, were inspected to identify unique features that differentiated fatigue behavior of dentin corresponding to the two age groups. The machined surface of particular beams was treated with dilute phosphoric acid solution (5% conc.) to remove the smear layer created by machining and enhance features on the tensile and compressive side of the beam.

3. Results

Typical responses resulting from monotonic flexural loading of the dentin specimens are shown in terms of the maximum bend stress and bend strain to failure in Fig. 2. In particular, Figs. 2a and 2b present representative responses from young (age = 17) and old (age = 77) patients, respectively. The flexural modulus was estimated from the bend responses (strain $\leqslant 0.4\%$) and is presented for each of the beams from the two age groups. A comparison of results from the young and old dentin indicated that age of the patient was an important factor. As evident in Fig. 2a, beams obtained from the younger patients exhibited linear elastic behavior at the onset of loading, followed by a region of nonlinear deformation to failure. In contrast, the beams of old dentin exhibited linear-elastic deformation to failure only (Fig. 2b). Beams obtained from patients of intermediate age (30 < age < 50; mean ± std. dev. = 39 ± 6) displayed evidence of nonlinear deformation but a lower strain to fracture than that of the young dentin.

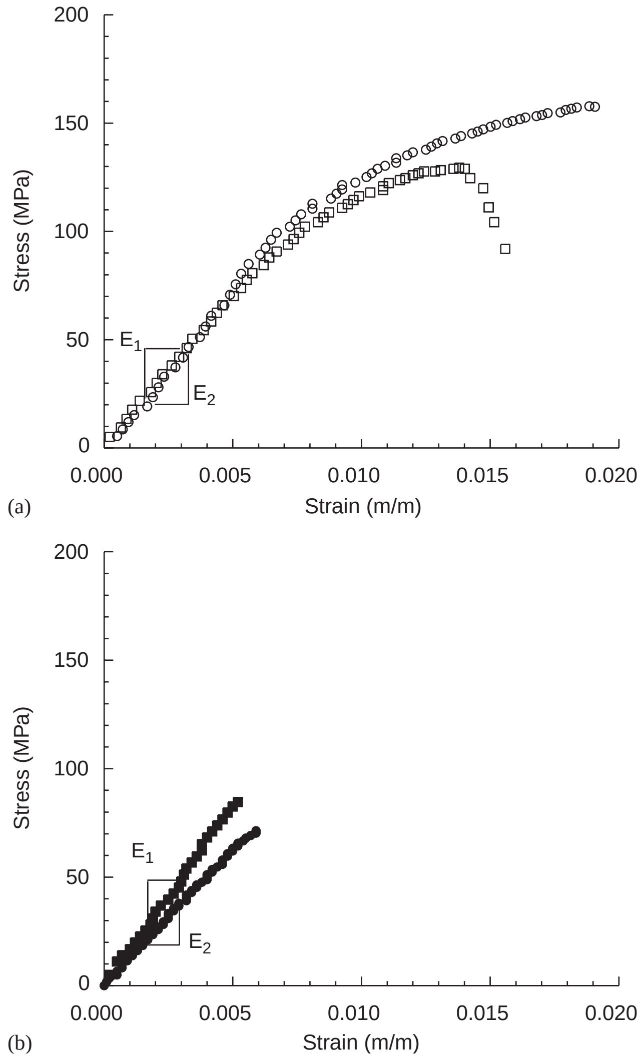

Fig. 2. Typical flexural response of the dentin beams under monotonic loading. The quantities E_1 and E_2 represent the elastic modulus of the respective specimens. (a) Young dentin: the beams were obtained from a 17-year-old female patient. $E_1 = 16.7$ GPa and $E_2 = 15.1$ GPa. (b) Old dentin: the beams were obtained from a 77-year-old female patient. $E_1 = 16.9$ GPa and $E_2 = 12.1$ GPa.

The distribution in flexural strength of dentin with age is shown in Fig. 3(a). The corresponding energy to fracture was distinguished from the area under the

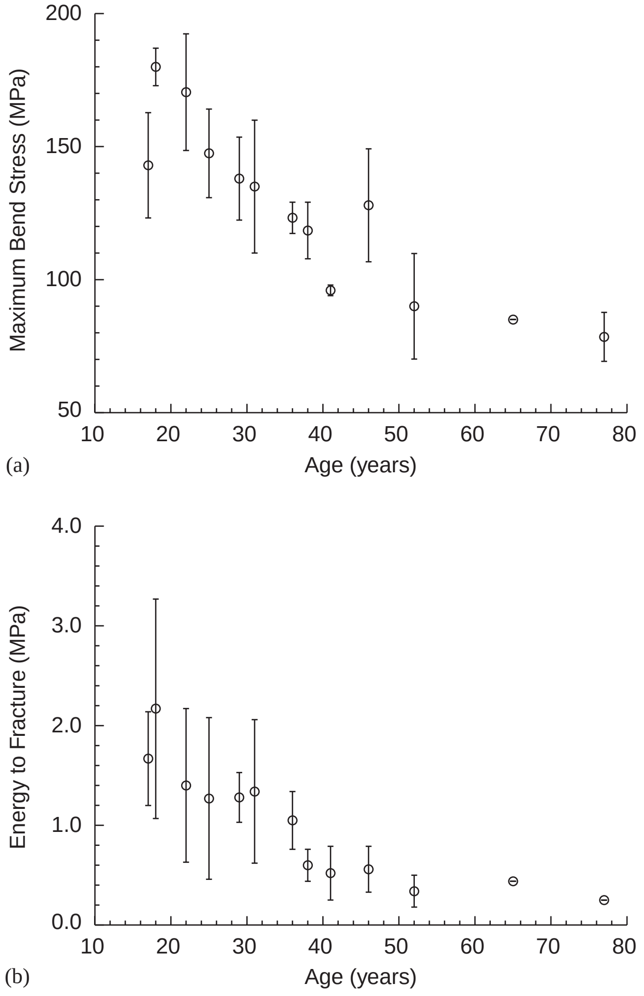

Fig. 3. Mechanical properties of dentin in terms of the patient age: (a) maximum bend stress, (b) energy to fracture.

flexural stress-strain curve and is shown in Fig. 3b. Each data point in Fig. 3 represents the average and standard deviation. There was a consistent reduction in the average bend strength and energy to fracture with age. A student *t*-test was used to compare properties of the young (age$\leqslant$30) and old (age$\geqslant$50) dentin according to the age distribution used in categorizing the fatigue specimens. The flexural strength of the young (mean age $=24\pm5$ years) dentin was significantly greater than that of the old (mean age $=65\pm13$ years) dentin ($p<0.0001$). Similarly, the energy to fracture of the young dentin was significantly higher than the old dentin ($p<0.0013$). There was no significant change in the flexural modulus of dentin with age.

In fatigue, the young dentin exhibited a component of nonlinear behavior that was evident from hysteresis in the load load-line displacement responses. In contrast, the older dentin generally behaved in a linear-elastic manner. Typical responses from young and old dentin specimens that resulted from fatigue loading are shown in Figs. 4a and b, respectively. The stiffness (S) of the dentin beams in fatigue was estimated from a least squares error estimate of the entire load load-line displacement response and the corresponding flexural modulus was estimated according to Eq. (2). Examples of the change in flexural modulus with fatigue that occurred with cyclic loading are shown in Fig. 5. Interestingly, beams from the young dentin (Fig. 5a) underwent an increase in stiffness and elastic modulus with cyclic loading until reaching a peak, after which the modulus decreased with further cyclic loading. According to the distinct transition in flexural modulus with cyclic loading, the responses were divided into two regions as shown in Fig. 5a. Furthermore, the flexural modulus was normalized by the maximum flexural modulus and the fatigue cycles were normalized by the fatigue life of each specimen. Typical cyclic responses for representative young and old dentin specimens are shown in Figs. 5b and c, respectively. Overall, the largest increase in stiffness, and the largest rate of change in stiffness, with fatigue resulted from specimens subjected to large cyclic stress amplitudes. In contrast to young dentin, there was a decrease in stiffness in the old dentin from the onset of fatigue loading (Fig. 5c). After further cyclic loading a rapid reduction of stiffness occurred that coincided with failure. In comparing responses from the two age groups the young dentin underwent the largest reduction in flexural modulus to failure with an average reduction between 15% and 20%. The average reduction in flexural modulus of the old dentin with cyclic loading to failure ranged between 5% and 10%.

Fatigue life diagrams for the young and old dentin are presented in Fig. 6a. Each data point corresponds to failure of a single dentin beam and data points with arrows identify beams that did not fail. As evident from Fig. 6 the fatigue strength of both young and old dentin decreased with cyclic stress amplitude. The young dentin exhibited a fatigue life approximately two decades greater than that of the old dentin at the same cyclic stress level, regardless of the stress amplitude. Power law models were developed to describe the mean fatigue response of each age group and are presented in Fig. 6b. The mean fatigue life distribution for each age group is also presented with a 95% confidence interval. A fatigue limit could not be defined definitively for either the young or old dentin. Using the power law model, the apparent endurance strength of young dentin at 10^7 cycles is approximately 44 MPa.The old dentin exhibited an apparent endurance strength at 10^7 cycles of approximately 23 MPa.

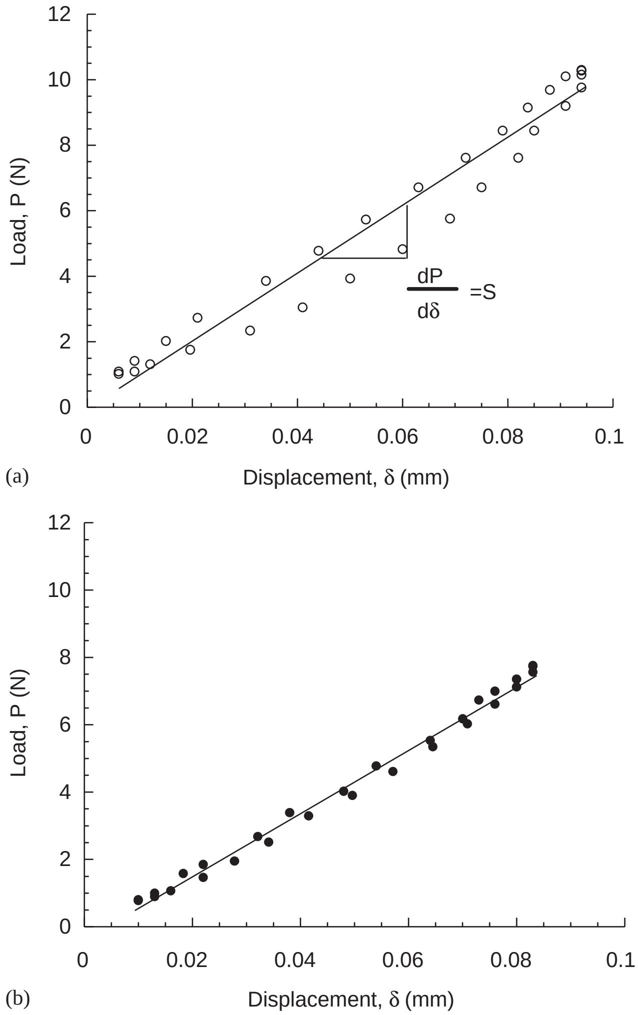

Fig. 4. Typical load load-line displacement responses for dentin resulting from four-point flexure: (a) young dentin, (b) old dentin.

There was a distinct difference in the fracture surface morphology between the young and old dentin specimens. Micrographs of the fracture surface from typical young and old dentin fatigue specimens are shown in Figs. 7a and b, respectively. The top of the beam

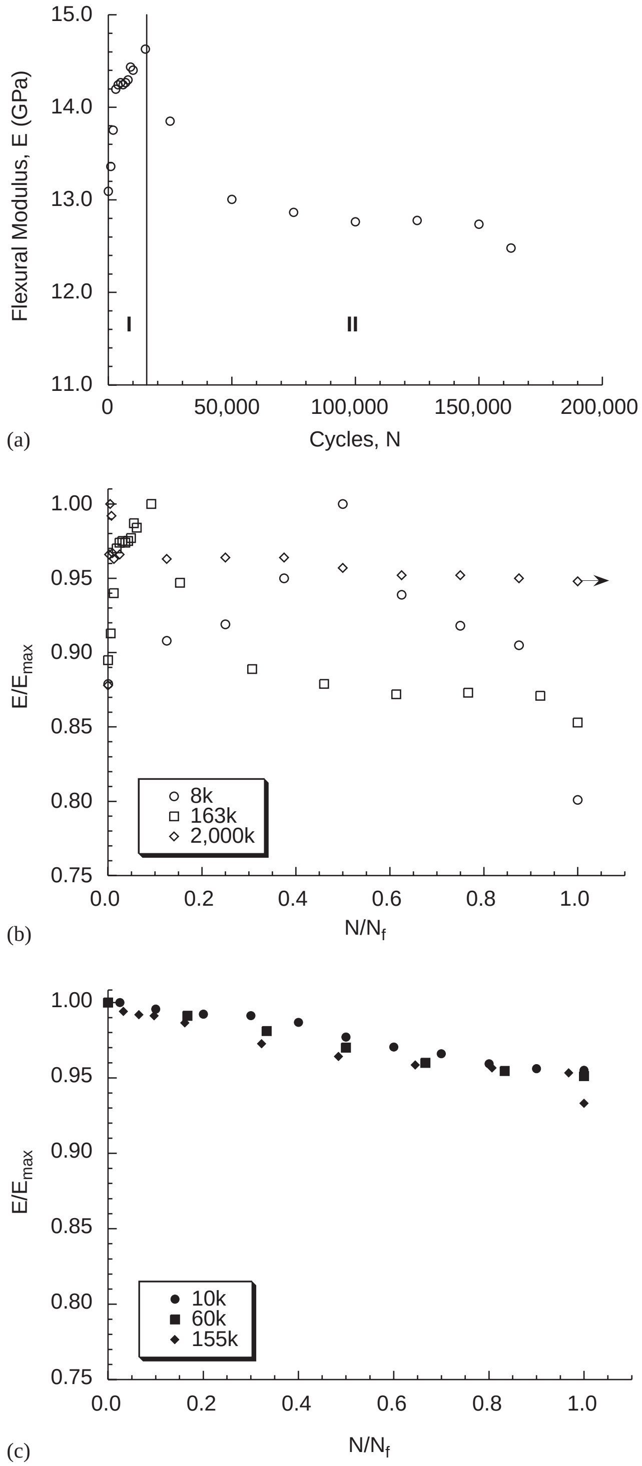

Fig. 5. Loss in flexural modulus with fatigue loading of the young and old dentin. The legend in each graph indicates the number of cycles to failure or the number of fatigue cycles if failure did not occur. (a) Typical stiffness history resulting from fatigue loading of young dentin illustrating two distinct regions of response. Patient age = 24 years. Stiffness loss between E_{max} and $E_{failure}$ ≈15%. (b) Stiffness loss for young dentin cycled at various stress amplitudes. Note that Region I behavior is most prevalent at higher stress amplitudes. (c) Stiffness loss for old dentin cycled at various stress amplitudes. Note the absence of Region I behavior.

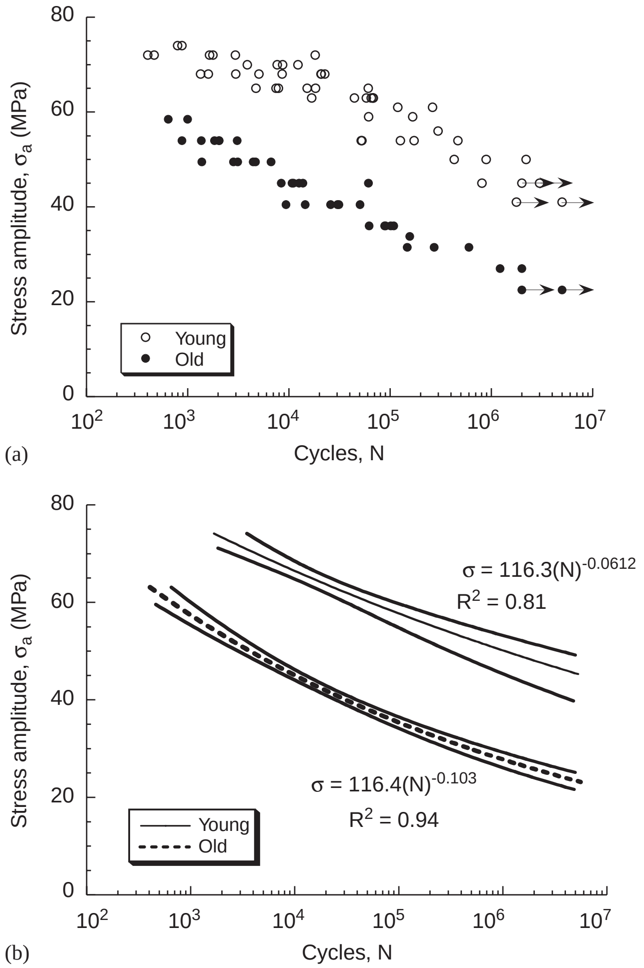

Fig. 6. Fatigue life diagrams of human dentin resulting from 4-point flexure loading with stress ratio (R) of 0.1. (a) Fatigue-life diagrams for young (mean age = 25 years) and old (mean age = 65 years) dentin. Arrows indicate specimens which did not fail at a particular number of cycles. (b) Power law model and 95% confidence interval for the mean fatigue responses of young and old dentin.

represents the tensile surface in each figure; the dentin tubules are oriented perpendicular to the length of the beam and are parallel with the majority of the fracture surface. Specimens of young dentin fractured perpendicular to the length of the beam and exhibited an overload shear lip on the compressive side of the neutral axis (Fig. 7a). An identification of a shear lip on the fracture surface of human dentin specimens has been documented previously in the fatigue fracture surface of cantilever beams [21]. However, in contrast to the young dentin, the old dentin specimens generally did not exhibit a shear lip (Fig. 7b). This quality is consistent with the behavior of a brittle material. One of the most notable differences between the young and old dentin was found when comparing microscopic features. Micrographs from the fracture surfaces of the young and old fatigue specimens in Fig. 7 are presented in Figs. 7c and d, respectively. A reduction in diameter of

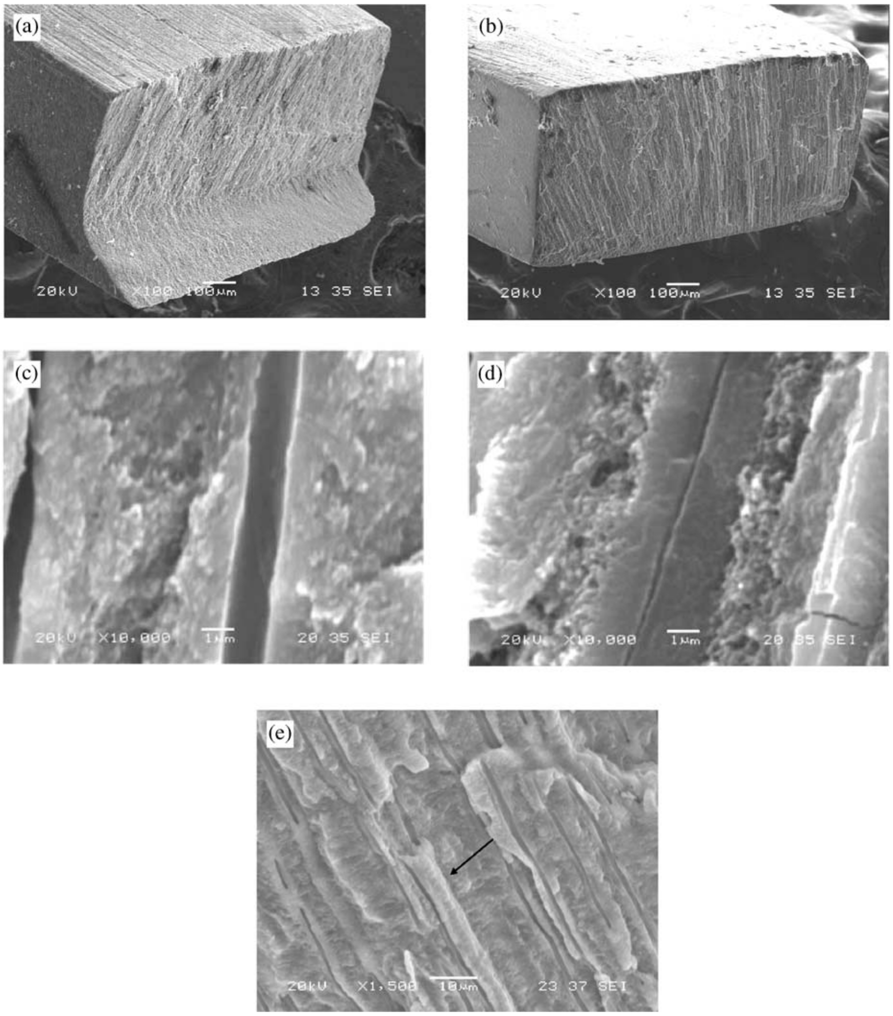

Fig. 7. Micrographs of the fracture surfaces from young and old dentin specimens. Micrographs (c), (d) and (e) were obtained from the tensile portions of the beams resulting from flexure loading. (a) A young dentin specimen (age = 20). Note the presence of a shear lip that developed at overload. (b) An old dentin specimen (age = 67). Note the absence of a shear lip. (c) Fracture surface of young dentin at high magnification. (d) Fracture surface of old dentin at high magnification. Note the difference in peritubular cuff thickness and lumen diameter. (e) A specimen from a 19-year-old patient. Note portion of fracture surface on the boundary of the peritubular dentin.

the tubule lumens was observed in specimens obtained from all of the old patients and the extent of occlusion increased with age. The differences in lumen and peritubular cuff dimensions with age are evident from a comparison of the micrographs for the young (Fig. 7c) and old (Fig. 7d) dentin specimens. Also of note, fatigue fracture in the young dentin often progressed through the peritubular cuff as well as along the boundary of the intertubular and peritubular dentin (Fig. 7e). However, fatigue fracture of the old dentin occurred more regularly through the tubules rather than along the intertubular/peritubular interface.

In an evaluation of fractured young and old dentin beams using the SEM, microcracks were observed on the tensile surfaces. As expected, the cracks were oriented parallel to the plane of maximum normal stress and the density of microcracks was greatest on the tensile side of the beams within the region of constant bend stress. Most cracks evident on the surface ranged in length from 10–150 μm. An electron micrograph of a young dentin sample with microcracks present on the tensile surface is shown in Fig. 8a. Note that the microcrack in this figure extends through individual tubules and along the interfaces of intertubular and peritubular dentin as previously noted. Interestingly, many of the microcracks in the young dentin specimens appeared to terminate within a hollow tubule as apparent in the micrograph presented in Fig. 8b. This observation implies that cracks progressed from one tubule to another and underwent temporary blunting within the tubule. In observing the beams of old dentin, very few microcracks were evident regardless of the cyclic stress amplitude. In addition, the microcracks appeared to propagate exclusively through the peritubular cuff rather than at the interface of intertubular and peritubular dentin as noted in the young dentin

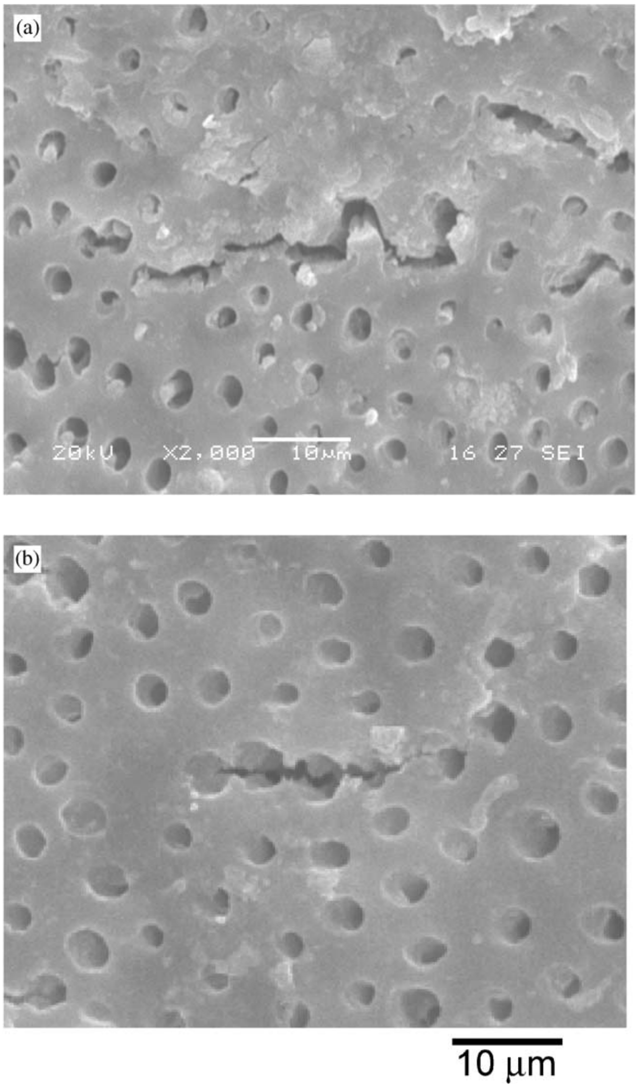

Fig. 8. Micrographs obtained from the tensile side of a beam of young dentin. The microcracks are oriented perpendicular to the orientation of maximum principal stress (a dilute phosphoric acid solution was used to remove the smear layer present on the machined surface). (a) Typical microcracks found on the tensile surface. (b) Microcrack extending between the tubules.

(Fig. 8a). Due to occlusion of the tubules, blunting of microcracks via the hollow tubule did not appear as a prominent mechanism of crack growth retardation in the old dentin. In comparison to features on the tensile side of the dentin beams there was no evidence of damage on the compressive side, regardless of age.

4. Discussion

According to fatigue life diagrams developed from results of the flexure experiments conducted with stress ratio (R) of 0.1 and load frequency of 5 Hz, the apparent endurance strength for the young ($17 \leqslant \text{age} \leqslant 30$, mean ± std. dev. = 25 ± 5 years) and old ($50 \leqslant \text{age} \leqslant 80$, mean ± std. dev. = 64 ± 9 years) dentin was 44 and 23 MPa, respectively. Nalla et al. [21] recently investigated the fatigue behavior of human dentin and reported that the apparent endurance strength of dentin at 10^6–10^7 cycles was approximately 25 and 45 MPa for frequencies of 2 and 20 Hz, respectively. It was later reported that the endurance strength of dentin at 10 Hz was reduced from approximately 30 to 20 MPa for fatigue loading with stress ratios of $R = 0.1$ and 0.5, respectively [22]. Although age of the molars used in the study was not reported, it would be expected that the molars were more likely from young patients ($17 \leqslant \text{age} \leqslant 30$). On that premise, the fatigue strength for young dentin from the present study is greater than that previously reported. Differences in results between these two studies are to be expected due to the tighter age distribution in the present study (and potential younger average age). There is also a difference in tubule orientation between the two studies. The tubules were parallel to the plane of the maximum normal stress in the present study, whereas the tubules were reportedly perpendicular to this plane in the previous study. Thus, the fatigue properties of dentin may be orientation dependent. There were also differences in the number of specimens examined. Nalla et al. [21,22] used approximately 10 specimens or less in constructing the fatigue life diagrams for lives between 10^3 and 10^6 cycles. Nevertheless, results from both studies confirm that the fatigue strength of dentin decreases with increasing cyclic stress amplitude.

The difference in stress-life responses for young and old dentin (Fig. 6) emphasizes the importance of aging on the fatigue properties of dentin. Tonami and Takahashi [20] previously investigated the effects of aging on the fatigue strength of bovine dentin and found the endurance strength to be 51.0 and 46.9 MPa for young and old (adult) bovine dentin, respectively. Note that the reported endurance strength was defined at 10^5 cycles, which is quite low considering that humans experience approximately 10^6 cycles of mastication in one year. Furthermore, the total age range was limited to approximately 4 years. Using the power law relationships for fatigue life of the human dentin (Fig. 6b), the mean fatigue strength estimated for the young and old dentin at 10^5 cycles is 58 and 38 MPa, respectively. These values agree reasonably well with results reported for the fatigue strength of bovine dentin. Although the difference in endurance strength between the young and old dentin reported by Tonami and Takahashi was significant, the stress ratio was not the same for both age groups ($R = 0.17$–0.25). In light of findings by Nalla et al. [22], this may have contributed to the interpreted difference in fatigue strength between the young and old bovine dentin. The fatigue life distributions for human dentin in Fig. 6b convey that there was a significant difference between the young and old dentin regardless of cyclic stress amplitude. Though the mean ages of the two groups was nearly 40 years apart, the absolute definition of old dentin was not established. Further investigation is necessary to distinguish the age-dependent fatigue strength of dentin and identify if there is a transitional age after which a significant reduction takes place.

The stress ratio used for the fatigue flexure experiments ($R = 0.1$) resulted in a non-zero mean stress. Using the Goodman model [24] to account for mean stress effects, the equivalent endurance strength for fully reversed loading (σ_e) of young and old dentin is 67 and 32 MPa, respectively. According to the distribution in flexural strength with age presented in Fig. 3, the ratio of endurance strength to ultimate strength (σ_{ult}) for the young and old dentin is approximately 0.5 and 0.38, respectively. Although dependent on many factors, the ratio σ_e/σ_{ult} for most engineering materials ranges from 0.30 to 0.60 [21]. The σ_e/σ_{ult} for dentin falls within this range, but the smaller ratio for old dentin emphasizes the increased sensitivity to fatigue. It is generally accepted that the total fatigue life of a material is a summation of the time required for initiation of a well-defined flaw (initiation life) and the time consumed in propagation of the flaw to a critical length (propagation life). Through use of load-control flexure, the propagation phase of fatigue life was minimized in the present study. Therefore, the difference in fatigue life between young and old dentin (Fig. 6) appears indicative of the difference in initiation life. Though not the only contributors, the population and size of intrinsic defects are important to the initiation phase of fatigue life. There is an increase in thickness of the peritubular cuff with age through deposition of mineral salts and occlusion of the dentin tubules [7]. According to the rule of mixtures [25], there is also an increase in mineral content of dentin with age per unit volume tissue. The deposition process may increase the population of intrinsic defects contributing to damage initiation in old dentin, thereby increasing the sensitivity to fatigue. Further work is underway to characterize defects in dentin and their contribution to fatigue and fracture.

Damage initiation and coalescence with fatigue and their contribution to the strength or stiffness of a material is an important component of structural behavior. In restorative dentistry, a reduction in stiffness or strength of the hard tissue foundation with cyclic loading is particularly relevant to the success of restorative practices. The degradation of either property can be quantified in terms of damage using three stages of response, namely, an initial steep decline, gradual deterioration and failure (e.g., [26]). An accumulation of microdamage induced by fatigue loading has been shown to reduce the stiffness and cause degradation in the mechanical properties of hard tissues [27,28]. In bone, recent studies have quantified the degradation of mechanical properties by monitoring the compliance, which originates from the development and growth of microcracks [29,30]. A similar approach may be appropriate for describing fatigue failure of human dentin. After an initial increase in flexural modulus in the young dentin specimens (Fig. 5a; Region I), there was a consistent decrease in modulus with further cyclic loading. The old dentin underwent a decrease in modulus from the onset of cyclic loading, but to a smaller extent than that for young dentin. In general, the young dentin underwent a decrease in flexural modulus of 10–20% prior to failure while the reduction for old dentin was limited to 5–10% (Fig. 5a). The reduction in modulus is expected to result from generation of microcracks with cyclic loading. A reduction in stiffness would also occur with development of a macrocrack and the stiffness loss in dentin could be perceived as a function of both microscopic and macroscopic damage. Note, however, that the experiments were conducted under load control fatigue and would result in very rapid growth of a macrocrack after coalescence. Differences in the flexural modulus history with cyclic loading for young and old dentin (Fig. 5) suggest that the old dentin is less tolerant to damage than young dentin. It is worthwhile to consider these findings in light of the microscopic analysis. Observations of the dentin fatigue specimens using the SEM showed that microcracks on the tensile surface of old dentin were sparse, while microcracks were apparent more frequently on the younger dentin specimens. The lower density of microcracks in the old dentin samples may be attributed to differences in the rate of damage initiation, but could also result from a larger rate of damage propagation in old dentin. Arola and Sundaram [31] recently studied the effects of age and dehydration on fatigue crack growth in human dentin where it was found that the average rate of fatigue crack growth within the steady-state growth regime (Region II) for old dentin (2.8E-4 mm/cycle) was more than a factor of magnitude greater than that for young dentin (1.2E-5 mm/cycle). It was also found that the stress intensity threshold (ΔK_{th}) of the old dentin was less than that for young dentin as well. Thus, aging appears to contribute to both the initiation stage of fatigue (via population of intrinsic defects) as well as the propagation phase (via rate of fatigue crack growth).

At present it is not clear what is responsible for the increase in stiffness of the young dentin specimens with cyclic loading. Zioupos et al. [32] recently reported that the fatigue behavior of bone has a time, strain-rate and cyclic dependency. Dentin has been reported to behave in a similar manner where the fatigue life of dentin was subject to frequency effects, especially at longer lifetimes and when evaluated in terms of cycles to failure [21]. However, frequency effects on fatigue life were not as apparent when plotted with regards to time to failure.

The mechanisms contributing to this time dependent behavior of dentin are not yet fully understood, but have been speculated to arise from the collagenous microstructure, which behaves in a viscoelastic manner [33]. The increase in flexural modulus of young dentin with cyclic loading (Figs. 5a, b) may result from viscoelastic deformation that arises in response to the non-zero mean stress. It appears that once the viscoelastic component of deformation has been exhausted, damage initiation causes a reduction in stiffness. The responses for old dentin (Fig. 5c) suggest that the viscoelastic component of deformation does not contribute to the fatigue response and that damage initiation begins at the onset of cyclic loading.

There were clear differences in the structure of the young and old dentin (Figs. 7c, d) and it is expected that occlusion of the dentin tubules is responsible for some of the differences in fatigue properties. An evaluation of the fracture surfaces suggests that the tubules contribute to the path of fracture in the young dentin as evident from the topography. There was evidence of fracture identified about the interface between the intertubular and peritubular dentin in the young dentin specimens (e.g., Fig. 7e). In comparison, the fracture surfaces of old dentin specimens displayed no evidence of intertubular/peritubular interfacial failure. This may indicate that the peritubular cuffs are more tightly bound to the intertubular dentin with age. Similarly, in a recent evaluation of fatigue crack growth in dentin it was found that pullout of peritubular cuffs did not appear on the fracture surface of old dentin indicating that some mechanisms of energy dissipation operating in young dentin were not present in fracture of the aged tissue [31]. Thus, it may be inferred that changes in structure of dentin with age promote a brittle response (e.g. Fig. 2b) and results in a decrease in strength (Fig. 3a) and energy to fracture (Fig. 3b). Though a recent study [34] has shown that the elastic modulus of dentin increases with an increase in mineral content, there was no significant change in the flexural modulus of human dentin noted with age. Changes in the structure with age also appeared to decrease anisotropy in the fracture behavior. In the young dentin a compression shear lip was identified that promoted fracture perpendicular to the tubules. And in an evaluation of fatigue crack growth in bovine dentin the authors regularly observed crack curving that enabled crack extension perpendicular to the tubules [35]. The old dentin beams did not exhibit a shear lip (Fig. 7b) and fracture occurred exclusively on the plane of maximum normal stress oriented parallel to the dentin tubules. The collagen fibrils have been suggested to play an important role in the fracture behavior of dentin [36] and are oriented in planes roughly perpendicular to the dentin tubules. Therefore, a reduction of the lumen diameter and increase in mineral content may not be the only contributions to the observed changes in mechanical behavior of human dentin with age. Changes in the collagen matrix may also occur and contribute to the structural response.

Though results of the investigation are of importance to the field of restorative dentistry, there are several recognized limitations. Previous studies have shown that the rate of fatigue crack propagation in dentin is a function of tubule orientation [31,35]. The initiation of fatigue damage in dentin and the stress-life response is expected to be orientation dependent as well. However, the experimental evaluation was limited to coronal dentin and a single tubule orientation. Additional study is currently underway to establish the influence of tubule orientation on the fatigue strength of human dentin. The experimental evaluation was conducted at ambient temperatures (22 °C) rather than at 37 °C. An earlier evaluation reported that the fracture properties of dentin are temperature invariant from 0 °C to 60 °C [37]. Therefore, differences in the flexural responses due to temperature are expected to be small. There are other factors that likely contribute to the fatigue life of dentin with age including frequency of loading, mean stress, hydration and various physiological factors. Thus, further research on the fatigue properties of dentin appears warranted. Nevertheless, this investigation has established that the fatigue properties of dentin are dependent on age and should be considered in the field of restorative dentistry in the pursuit of lifelong oral health.

5. Conclusion

An experimental evaluation of the effects of age on the fatigue properties of human dentin was conducted. Rectangular beams were prepared from the coronal dentin of virgin molars with the dentin tubules oriented perpendicular to the long axis of the beams. The specimens were loaded in either quasi-static 4-point flexure or under 4-point flexure fatigue at 5 Hz and stress ratio (R) of 0.1. Based on results from this study the following conclusions were drawn:

1. The maximum flexure strength and energy to fracture of dentin decreases with age. The mean flexural strength of dentin beams from the youngest patients (17) exceeded 140 MPa, whereas dentin beams from the oldest patients exhibited a mean strength of less than 80 MPa.
2. There is a reduction in the fatigue strength of dentin with age. The apparent endurance strength (defined at 10^7 cycles) of the young ($17 \leqslant \text{age} \leqslant 30$, mean ± std. dev. = 25 ± 5 years) and old ($50 \leqslant \text{age} \leqslant 80$, mean ± std. dev. = 64 ± 9 years) dentin was found to be approximately 44 and 23 MPa, respectively.

3. The fracture surfaces of young dentin exhibited a large overload shear lip. The old dentin specimens exhibited little or no such feature. The difference in fracture surface morphology and flexural response to failure indicates that dentin becomes more brittle with age.
4. The old dentin was less tolerant to damage than young dentin. Specimens of young dentin underwent a decrease in flexural modulus to failure of 10–20%. The reduction in modulus of old dentin with fatigue was 5–10%. At the onset of fatigue loading young dentin underwent an increase in modulus until reaching a peak, after which a reduction in modulus occurred to failure. The flexure modulus of the old dentin decreased from the onset of fatigue loading.
5. Microcracks were observed on the tensile side of the dentin beams and ranged in size from 10–150 μm. Microcracks were more prevalent in young dentin and provided evidence of an increased ability to withstand fatigue damage. Based on differences in the stiffness history and microcrack density, aging appears to result in an increase in both the rate of damage initiation and propagation in dentin.

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