# Hydration and dynamic fatigue of dentin

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**Abstract:** An experimental investigation on the dynamic fatigue response of dentin was conducted to examine the influence of stress rate on the strength and energy to fracture. Rectangular beams were prepared from the coronal dentin of bovine maxiallary molars and subjected to four-point flexure to failure. The dentin beams were examined in the fully hydrated and dehydrated condition at stress rates ($\dot{\sigma}$) ranging from 0.01 to 100 MPa/s. Results for the hydrated dentin showed that the flexure strength, energy to fracture, and flexure modulus all increased with increasing stress rate; the flexure strength increased from 100 MPa (($\dot{\sigma}$) = 0.01 MPa/s) to 250 MPa (($\dot{\sigma}$) = 100 MPa/s). In contrast, the elastic modulus and strength of the dehydrated dentin decreased with increasing stress rate; the flexural strength of the dehydrated dentin deceased from 170 MPa (($\dot{\sigma}$) = 0.01 MPa/s) to 100 MPa (($\dot{\sigma}$) = 100 MPa/s). While the hydrated dentin behaved more like a brittle material at low stress rates, the strain to fracture was found to be nearly independent of ($\dot{\sigma}$). According to the experimental results, restorative conditions that cause development of static stresses within the tooth could promote a decrease in the damage tolerance of dentin. © 2005 Wiley Periodicals, Inc. J Biomed Mater Res 77A: 148–159, 2006

**Key words:** dentin; fatigue; fracture; hydration; restoration failure

## INTRODUCTION

Dentin is a hydrated hard tissue that comprises the majority of the human tooth by both weight and volume.[1] A thorough understanding of the mechanical behavior of dentin is required for the development of the next generation of restorative procedures and in the design of new materials for tissue replacement. Knowledge of the mechanical behavior of dentin is also essential for understanding the potential influences from oral diseases and aging on the pursuit of lifelong oral health.

Previous studies on the mechanical behavior of dentin have identified that the tissue exhibits time-dependent elastic (i.e. viscoelastic) behavior. In an early evaluation of hydrated dentin using compression tests, Craig and Peyton[2] found that a relaxation in stress occurred over time. Later, Korostoff and coworkers[3,4] quantified the relaxation modulus of hydrated radicular human dentin and found that the relaxation modulus decreased exponentially with time and that beyond a threshold time, the relaxation was negligible. More recently Balooch et al.[5] evaluated the properties of fully hydrated demineralized human dentin using atomic force microscopy (AFM) and identified that the viscoelastic behavior was independent of location. Kinney et al.[6] also used AFM in evaluating the elastic properties of human dentin and noted that relaxation was potentially a function of hydration. The elastic modulus reported for dentin ranges from ~10 GPa to nearly 30 GPa; this range is believed largely attributed to the influence of strain rate effects and viscoelastic response.[7] Indeed, Jantarat et al.[8] showed that the elastic modulus of human root dentin increased with an increase in strain rate.

Although the mechanisms responsible for time-dependent relaxation are not completely understood, evaluations of fully mineralized and demineralized dentin suggest that the viscoelastic behavior is potentially attributed to the movement of water and properties of the collagen fibril network.[5–7,9] There are structural changes that occur to the collagen fibrils with dehydration.[10,11] These changes are of substantial importance to the hybrid layer morphology and resin/dentin bonding. But hydration is also highly relevant to the mechanical behavior of dentin. Trengrove et al.[12] noted that stress relaxation in human dentin decreased after air-drying in comparison to fully hydrated dentin. Dehydration has been found to cause an increase in the elastic modulus and ultimate

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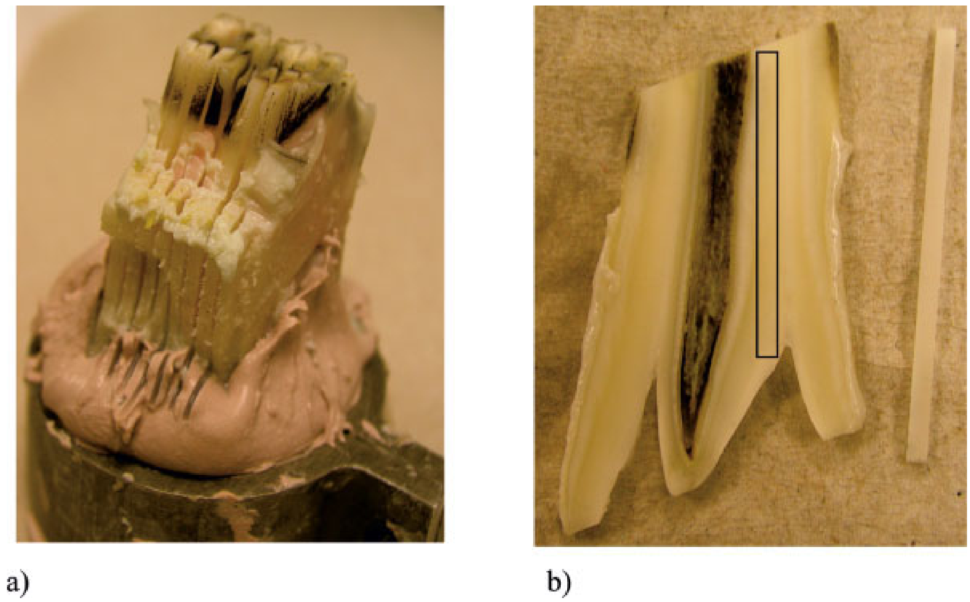


**Figure 1.** Preparation of flexure specimens from a bovine molar for dynamic fatigue: (a) a molar with a series of sections along the buccal-lingual axis and (b) potential flexural specimens. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

strength of dentin.[13] A decrease in moisture content also reportedly results in a reduction in the strain to fracture and fracture toughness.[14–16] According to the effects of strain/stress rate and moisture on the elastic behavior of dentin, it would appear reasonable that the inelastic components of mechanical behavior are also rate- and moisture-dependent.

In this investigation, the mechanical behavior of dentin was evaluated with respect to rate of loading and hydration according to a dynamic fatigue analysis. The primary objective was to identify the influence of stress rate and hydration on the strength and energy to fracture of dentin.

## MATERIALS AND METHODS

Dentin is considered a hard tissue that is ~40–45% inorganic, 35% organic, and 20–25% water by volume.[1] The organic and inorganic components are comprised of a collagen fibril network and apatite crystallites, respectively. There is a network of tubules in both bovine and human dentin that extend from the pulp outwards towards the dentin-enamel junction (DEJ). The tubules exist as open channels (1–2 μm internal diameter), which are surrounded by a hypermineralized cuff of tissue referred to as the peritubular dentin. Bovine dentin was used as a model for human dentin in this study based on similarities in tissue structure[17,18] and availability. In bovine dentin, the peritubular cuff surrounding each tubule is between 0.5 and 1 μm in wall thickness, which is consistent with that of human dentin. Though the microstructure of human and bovine dentin is quite consistent, the density of tubules in bovine molars is lower. A recent evaluation of fatigue crack growth in the dentin of bovine molars reported a tubule density from 1700 to 20,000 tublues/$mm^2$,[19] while the tubule density for human coronal dentin generally exceeds 20,000 tubules/$mm^2$.[1]

Fully erupted bovine maxillary molars were obtained from mature animals (between age of 1 and 3 years old) within 12 h of slaughter. No details were available on the specific age, sex, health, or breed of the animals. The molars were extracted by sectioning the jaw and then visually inspected for caries and flaws. Those without damage or decay were immediately stored in a saline medium at 2°C to maintain hydration. The storage solution consisted of 900 mg/L NaCl buffered with 57.5 mg/L $CaCl_2$ according to Gustafson et al.[20]; the resulting pH of the solution was 6.0. Selected molars were potted within a polymeric foundation (Fig. 1 and then sectioned using a numerically controlled slicer/grinder (K.O. Lee Model S381EL, Aberdeen, SD) with diamond-impregnated slicing wheels (No. 220 and No. 320 mesh abrasives) and water-based coolant. The crown of the molars was removed first to identify areas that are likely to provide sound dentin. Sections were then introduced along the bucco-lingual axis to obtain dentin slices of ~1.5 mm thickness [Fig. 1(a)]. The slices were bonded to a glass plate and rectangular beams were then machined from the individual sections according to Figure 1(b). All edges of the beams were lightly dressed using No. 320 grit abrasive papers to smooth any rough edges. A schematic diagram of the final specimen geometry is shown in Figure 2(a). In general, three or fewer molars were obtained from each cow and less than ten specimens were obtained from each molar. All of the molars used in this study were obtained from eight different animals.

The influence of rate of loading and hydration on the mechanical behavior of dentin was evaluated according to a dynamic fatigue analysis. Dynamic fatigue is a term used interchangeably with slow crack growth (SCG) and is used

**TABLE I**
**Summary of the Number of Dentin Specimens Tested at Each Load and Stress Rate**

| | Load Rate, N/s (Stress Rate, MPa/s) | | | | |
|---|---|---|---|---|---|
| Material | 0.001 (0.01) | 0.01 (0.1) | 0.1 (1) | 1 (10) | 10 (100) |
| Hydrated dentin | 12 | 16 | 12 | 11 | 11 |
| Dehydrated dentin | 5 | 5 | 5 | 5 | 5 |
| Glass | 5 | 5 | 5 | 5 | 5 |

in describing conditions that facilitate crack extension or fracture under a monotonic load applied at a constant stress rate. Slow crack growth is of relevance when the apparent stress intensity is much smaller than the fracture toughness.[21] Many ceramic materials exhibit a tendency for subcritical crack extension in aqueous conditions under static or quasi-static loads as a result of corrosion.[22] In the present study, dynamic fatigue experiments were conducted according to ASTM C1368[21] and ASTM C1161.[23] The slow crack growth parameters of engineering ceramics are generally determined using constant stress-rate flexural testing, and this approach was adopted to quantify the dynamic fatigue response of dentin. A four-point flexural arrangement with 1/3 load span was used for flexural loading [Fig. 2(b)] with a support span (L) of 6 mm. Consistent with the standard, the specimens were subjected to flexure loading at six different load rates that ranged from 0.001 to 10 N/s. A summary of the number of specimens tested at each of the load ranges is shown in Table I. To reduce the potential for bias that may be attributed to a specific molar or animal, the bend specimens from each tooth were evaluated at different stress rates. The beams were loaded under load control actuation until fracture, using an EnduraTEC ELF 3200 universal testing system. Specimens evaluated in the hydrated condition were loaded while immersed in a saline bath. Dehydrated dentin specimens were prepared by placing the specimens in a free convection laboratory oven at 60°C for ~24 h prior to the evaluation. The specimens were periodically weighed until the mass reached steady state after which the flexural loading was conducted. All specimens were tested within ~3 weeks of the day of slaughter.

Throughout each experiment, the displacement and load were acquired using the data acquisition system of the ELF 3200 using a rate of acquisition between 0.2 and 50 Hz that was scaled according to the loading rate. In an effort to validate the experimental procedures, flexure specimens were prepared from glass and tested using the aforementioned procedures and range of stress rates. The evaluation of glass was conducted at room temperature (22°C) in air. Glass was selected as a benchmark material for its inability to undergo plastic deformation and consequent lack of appreciable stress rate dependence. In addition, glass exhibits a flexure strength that is similar to that of dentin. A total of five glass specimens were examined at each of the five stress rates as indicated in Table I.

The maximum normal stress resulting from flexural loading was determined using conventional beam theory in terms of the bending moment, beam geometry, and moment of inertia. For a beam in four-point flexure with 1/3 point load span, the maximum stress between the two interior load points is given by

$$\sigma = \frac{PL}{bh^2} \quad [\mathrm{MPa}] \tag{1}$$

where $P$ is the transverse load, $L$ is the outer support span length, $b$ is the specimen width, and $h$ is specimen thickness. The flexural strength ($\sigma_f$) of each beam was determined from the maximum flexural load that was recorded. The stress rate (($\dot{\sigma}$)) for flexural loading can be described by

$$\dot{\sigma} = \frac{\dot{P}L}{bh^2} \quad [\mathrm{MPa/s}] \tag{2}$$

where $\dot{P}$ is the loading rate. The flexural strength of the dentin and influence of stress rate was modeled using the common power law relationship according to

$$\sigma_f = D\dot{\sigma}^{\,1/n+1} \quad [\mathrm{MPa}] \tag{3}$$

where $n$ and $D$ are the slow crack growth exponent and coefficient, respectively. The empirical parameters in Eq. (3) were determined by performing a power law least square regression fit of the flexural strength distribution plotted in terms of the stress rate.

Although generally defined from tensile testing, the specific energy to fracture of each dentin beam was also determined as a function of stress rate from the area under the bend stress–bend strain curves. The load and displacement acquired during the experiments were used to calculate the stress and strain distribution in the beam, respectively. The bend stress was calculated using Eq. (1) in terms of the specimen geometry, and the bend strain was estimated from the radius of curvature of the beam. Using the bend displacement (δ) recorded during the flexure experiments and geometry of the four-point load arrangement (support and load span as well as the beam geometry), the radius of curvature (ρ) was estimated according to

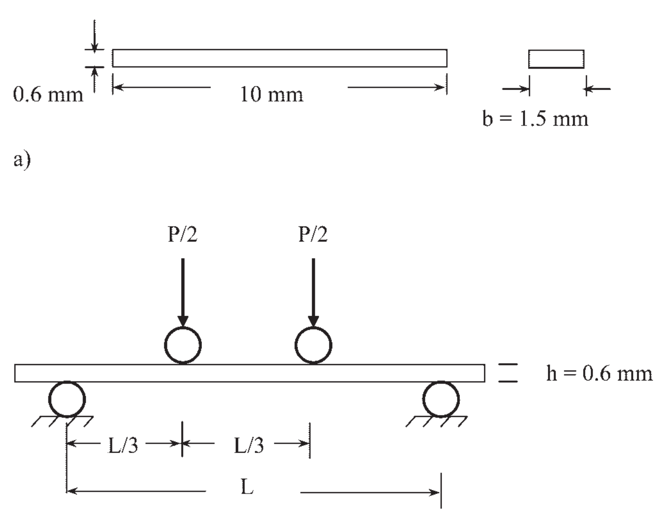


**Figure 2.** Schematic diagrams of the specimen geometry and load arrangement: (a) the flexure specimen geometry and (b) the four-point load configuration and 1/3 load spacing.

$$\rho = 4E-6 \cdot \frac{[(15.63E9 \cdot \delta^4 + 3.13E5 \cdot \delta^2 + 1)]^{0.5}}{\delta} \quad [m] \quad (4)$$

The normal strain resulting from flexure loading was estimated by dividing the distance from the neutral axis to the beam's surface (y) by the radius of curvature (ρ). Using both the stress and strain responses from the flexure experiments, the specific energy to failure of the dentin beams was estimated from the area under the bend stress - bend strain curve. A comparison of the elastic modulus, strength, and energy to fracture was conducted using Student's *t*-test. An evaluation of the change in dependent variables as function of the stress rate was conducted using the F-test.

Surfaces of the fractured dentin beams were evaluated using a Jeol 5600 scanning electron microscope (SEM) to identify the dentin tubule orientation with relation to the fracture surface and to understand differences in the dynamic fatigue response attributed to stress rate or hydration. The fractured specimens were sputtered with gold palladium to enhance conductance of the fracture surface and then examined in secondary electron imaging mode.

## RESULTS

A typical load, load-line displacement response obtained for flexural loading of a hydrated dentin beam at a stress rate of 10 MPa/s is shown in Figure 3(a); the corresponding stress–strain response at the surface of the beam is shown in Figure 3(b). Through a comparison of responses over the range in stress rates examined, it was noted that the load at fracture was largely dependent on the stress rate. Over the entire stress range from 0.01 to 100 MPa/s, the specimens failed at a maximum flexural load between 10 and 30 N. The experiments ranged in duration from over 4 h at the lowest stress rate to ~3 s at the largest stress rate.

The stress–strain responses for flexural loading at stress rates of 0.01, 1, and 100 MPa/s are shown in Figure 4(a–c), respectively. As evident from a comparison of the responses in this figure, the strength of the dentin beams was dependent on the stress rate. In general, the specimens exhibited a distinct linear and apparently elastic region, followed by a region of nonlinear deformation. Specimens loaded at the high stress rates appeared to exhibit the largest extent of nonlinear deformation. Overall, there was a larger variation in the stress–strain responses resulting from loading at the higher stress rates than at the lower stress rates.

The dynamic fatigue behavior of dentin after dehydration was also evaluated through an examination of the flexural strength in terms of the stress rate. Approximately 25 specimens were used for the five stress rates ranging from 0.01 to 100 MPa/s. Representative bend stress–bend strain curves for dehydrated dentin obtained at stress rates of 0.01, 1, and 100 MPa/s are shown in Figure 5(a–c), respectively. All of the dehydrated specimens exhibited a linear (and potentially elastic) response to failure, regardless of the rate of loading. In contrast to the hydrated dentin, there was little evidence of nonlinear deformation evident in the load, load-line displacement responses.

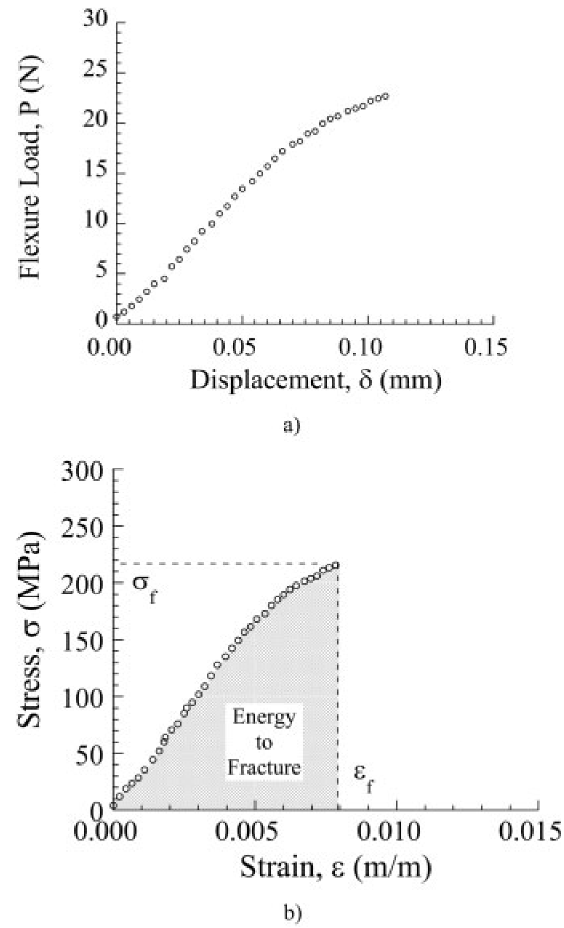


**Figure 3.** Flexural response under four-point loading (stress rate = 10 MPa/s): (a) load, load-line displacement and (b) stress–strain response.

The flexural strength for the hydrated and dehydrated dentin is plotted in terms of the stress rate in Figure 6(a). Each data point represents the average strength and standard deviation. Hydrated bovine dentin underwent a significant increase in flexural strength with increasing stress rate ($p < 0.01$). In contrast to the hydrated dentin, there was a significant decrease in the flexural strength of the dehydrated dentin with increasing stress rate ($p < 0.01$). In addition, a comparison of the responses showed that the hydrated dentin was significantly stronger ($p < 0.0001$) than the dehydrated dentin for ($\dot{\sigma}$) > 0.3 MPa/s (i.e. 1, 10, and 100 MPa/s). Utilizing the power law responses of the experimental results [Fig. 6(a)], the dynamic fatigue parameters were determined for the dentin in each of the two levels of hydration according to Eq. (3) and are listed in Table II. The slow crack growth exponent (n) of the hydrated dentin

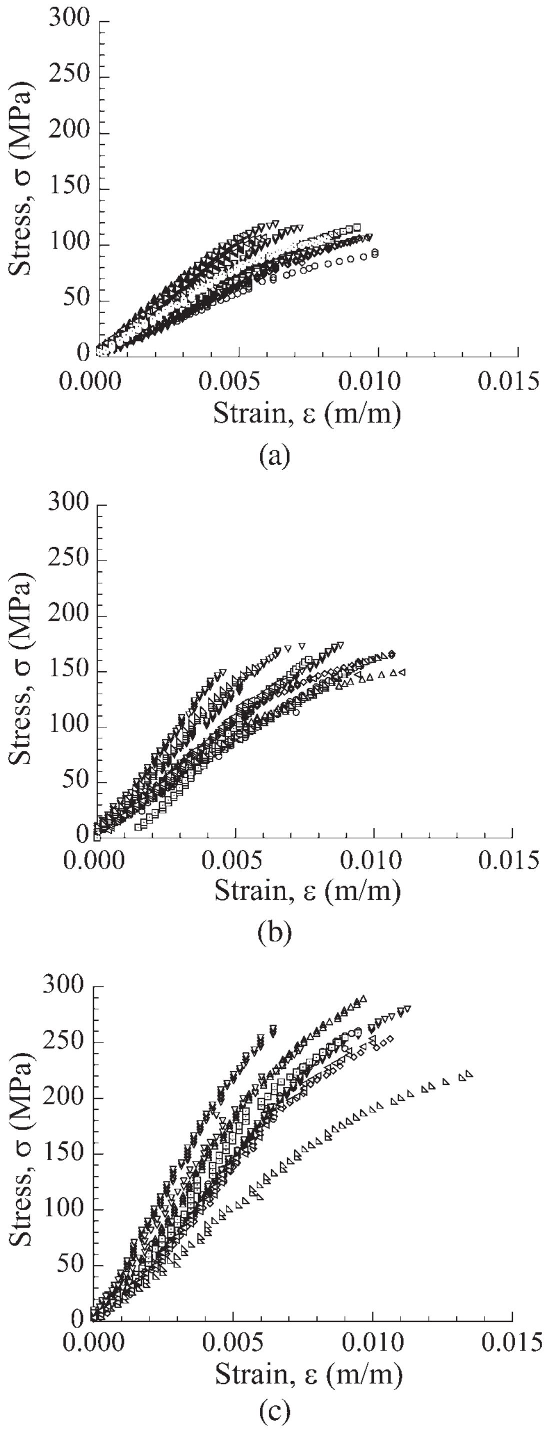


**Figure 4.** Flexure responses for the hydrated dentin beams: (a) stress rate of 0.01 MPa/s, (b) stress rate of 1 MPa/s, and (c) stress rate of 100 MPa/s.

specimens was 9 while the dehydrated dentin exhibited a negative exponent of larger magnitude (−21). Results from the flexure tests with glass indicated that the average strength was 85 MPa and essentially independent of stress rate ($n$ = 199). The n for glass was a factor of magnitude greater than that of the dentin, as expected, based on the relative independence to stress rate.

In addition to an evaluation of strength, the elastic modulus was also estimated from the flexure responses of the dentin specimens and is plotted in terms of the stress rate in Figure 6(b). For both hydrated and dehydrated specimens, the apparent elastic modulus in flexure was estimated using the data for strains less than 0.004 m/m. As evident

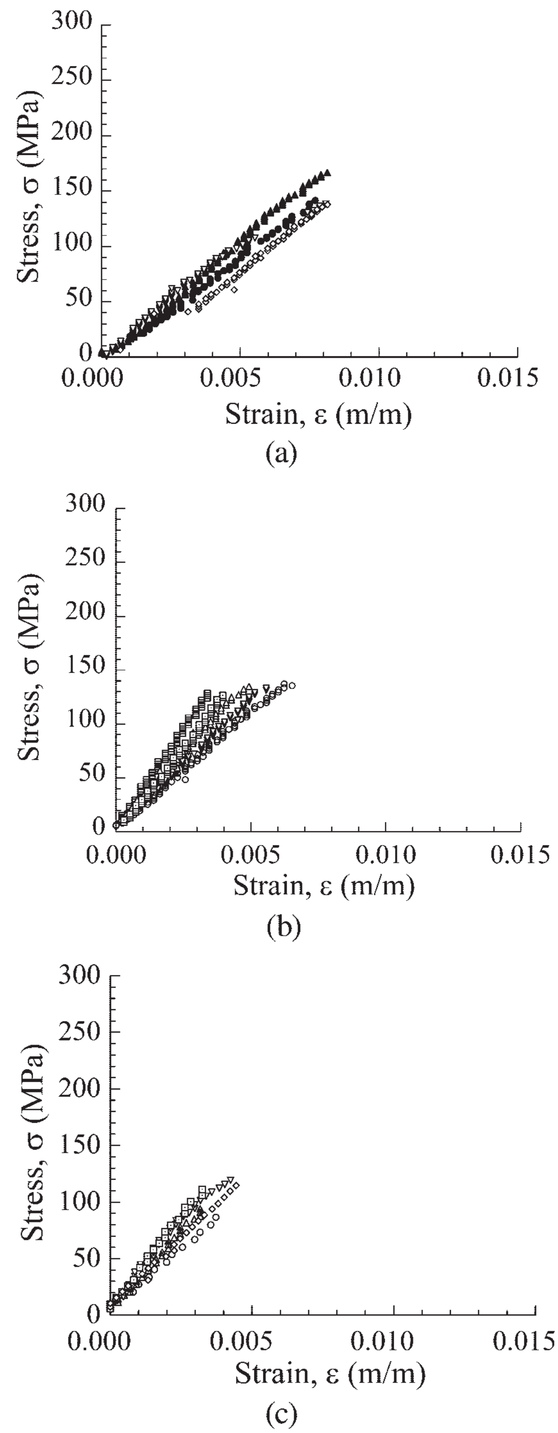


**Figure 5.** Typical flexure responses of the dehydrated dentin beams: (a) Stress rate of 0.01 MPa/s, (b) Stress rate of 1 MPa/s, and (c) Stress rate of 100 MPa/s.

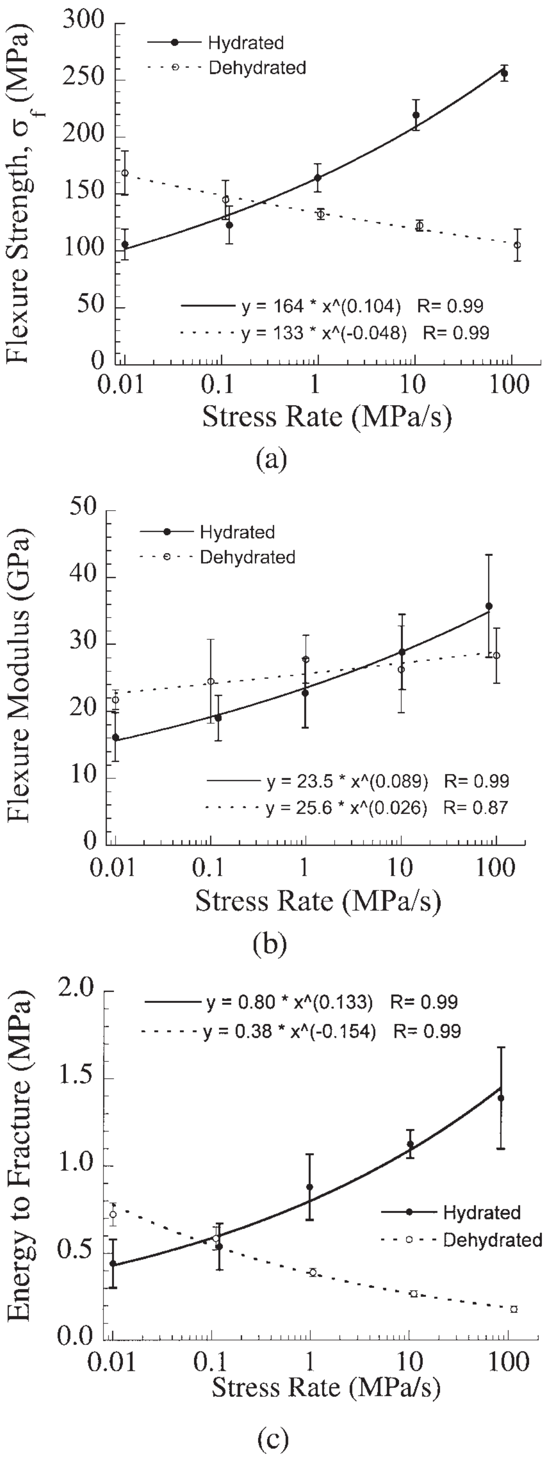


**Figure 6.** The influence of stress rate and hydration on the mechanical properties of dentin: (a) flexure strength, (b) flexure modulus, and (c) energy to fracture.

from the distribution in Figure 6(b), there is a significant ($p < 0.01$) increase in the flexural modulus of the hydrated dentin with increasing stress rate. Although there was no distinct trend, it appears that the flexure modulus of the dehydrated dentin also increased with stress rate. Interestingly, there was no significant difference in the flexure modulus of the hydrated and dehydrated dentin for all stress rates except at the lowest stress rate. At the stress rate of 0.01 MPa/s, the flexure modulus of the dehydrated dentin was significantly greater than that of the hydrated dentin ($p = 0.005$).

The energy to fracture of the dentin beams was estimated by integrating the area under the flexural stress–strain curves (e.g. Fig. 3) to the strain at fracture. This measurement of energy can be considered the flexural toughness and is plotted in terms of the stress rate in Figure 6(c) using experimental results for both the hydrated and dehydrated dentin. The hydrated dentin exhibited a significant rise in fracture energy with stress rate ($p < 0.01$), an increase of nearly 0.20 MPa per order of magnitude increase in stress rate. In contrast, the energy to fracture of the dehydrated dentin decreased significantly ($p < 0.01$) with increasing stress rate. Differences in the energy to fracture between the hydrated and dehydrated dentin were significant ($p \leq 0.007$) at all stress rates except for $(\dot{\sigma}) = 0.01$ MPa/s.

Fracture surfaces of both the hydrated and dehydrated dentin beams were evaluated using the SEM to identify the tubule orientation and differences that may have contributed to the mechanical behavior. Typical fracture surfaces from hydrated dentin specimens subjected to low and high stress rates are shown in Figure 7(a,b), respectively. On the basis of the locations in which the specimens were obtained from the molars, the dentin tubules were typically oriented perpendicular to the length of the beam and nearly perpendicular to the neutral axis (i.e., the tubules were approximately parallel to the plane of maximum normal stress). As evident in Figure 7(a), the hydrated beams subjected to flexure at low stress rates fractured along the plane of maximum normal stress. The fracture surfaces were primarily flat and relatively smooth, both features that are indicative of brittle failure. However, in examination of specimens that failed under flexure at higher stress rates (e.g. $(\dot{\sigma}) \geq 1$ MPa/s), a shear lip was often identified on the compressive side of the beams as shown in Figure 7(b). The plane of fracture progressed from an orientation nearly parallel to the tubules (on the tensile side of the beam) to one nearly perpendicular to the tubules (on the compressive side). In addition, the fracture surface of

**TABLE II**
**Slow Crack Growth Parameters Estimated from the Dynamic Flexure Strength of Bovine Dentin and Glass**

| Material | D (MPa) (MPa/s)$^{-n}$ | $n$ |
|---|---|---|
| Hydrated dentin | 162 | 9 |
| Dehydrated dentin | 134 | −21 |
| Glass | 85 | 199 |

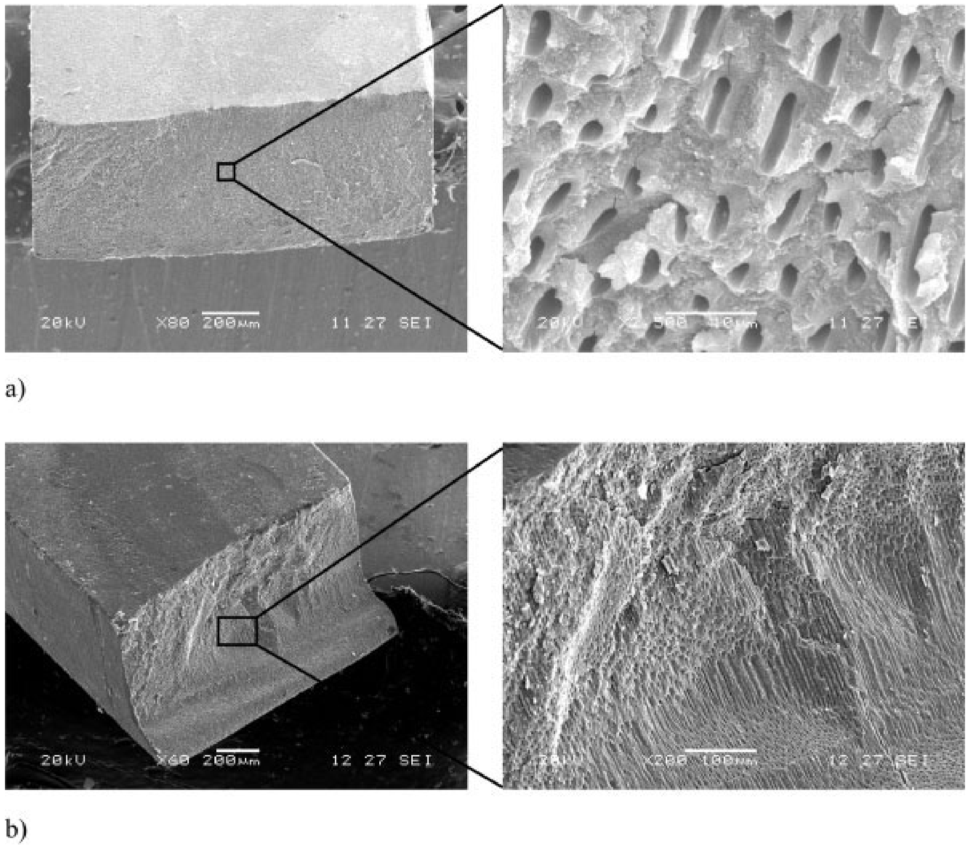


**Figure 7.** Typical fracture surfaces of hydrated dentin beams resulting from flexure. The top of the beam is the tensile surface: (a) fracture surface from a beam that failed at a low stress rate (0.01 MPa/s) and (b) fracture surface from a beam that failed at a high stress rate (100 MPa/s).

specimens loaded at high stress rates appeared to be more irregular when compared with those loaded at lower stress rates as evident from a comparison of Figure 7(a,b). Fracture surfaces of the dehydrated dentin specimens were also examined and surfaces from beams that failed at low and high stress rates are shown in Figure 8(a,b), respectively. While microsopic features of the fracture surfaces from the hydrated and dehydrated dentin appeared similar, some differences were evident from the macroscopic features. There was minimal evidence of a shear lip on the dehydrated dentin beams, regardless of stress rate. The fracture surfaces on both the tensile and compressive sides of the beam were oriented parallel to the plane of maximum normal stress, suggesting that failure occurred in a brittle manner and was not influenced by the tubule orientation.

## DISCUSSION

An experimental evaluation of the dynamic fatigue response of dentin was conducted and the influence of rate of loading and hydration on the mechanical behavior was examined. The stress rate or strain rate that arises from mastication is undoubtedly a function of the biting force, location in the tooth, whether the tooth contains a restoration, shape and size of the restoration, etc. Using results of a simple numerical analysis of the stress distribution in virgin and restored teeth,[24] the stress rate resulting from mastication would range from 0 to 40 MPa/s. Using the rate-dependent flexure modulus [Fig. 6(b)], a stress rate of 10 MPa/s would correspond to a strain rate of $\sim 5 \times 10^{-4}\ s^{-1}$. An experimental evaluation of strain rates in the cervical region during normal oral functions revealed strain rates ranging from $5 \times 10^{-4}$ to $9 \times 10^{-3}\ s^{-1}$.[8] The experimental evaluation of bovine dentin was conducted over stress rates ranging from 0.01 to 100 MPa/s, which corresponds to strain rates from $\sim 6 \times 10^{-7}$ to $4 \times 10^{-3}\ s^{-1}$. Thus, the experimental evaluation encompassed the rate of loading that arises in typical oral functions.

The flexure strength, flexure modulus, and energy to fracture of the hydrated dentin all increased significantly with increasing stress rate [Fig. 6(a–c)]. In particular, the flexure modulus of hydrated dentin increased from 16 to 35 GPa with increase in stress rate from 0.01 to 100 MPa/s. According to a recent review,[7]

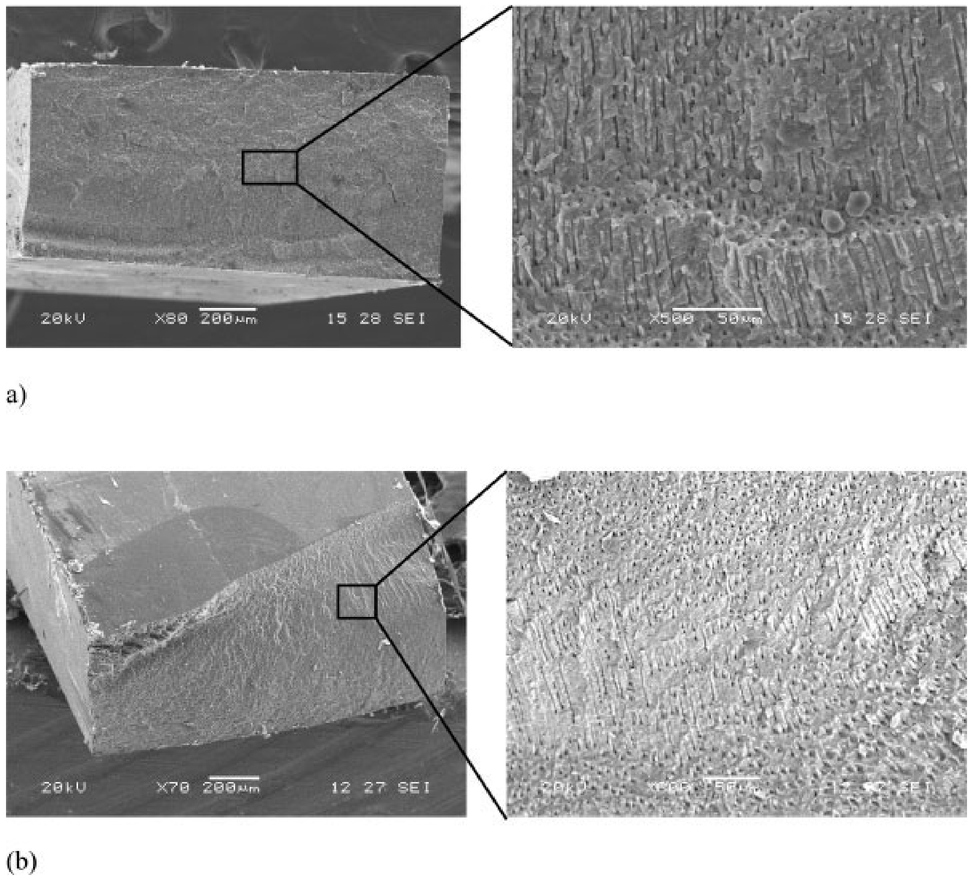


**Figure 8.** Typical fracture surfaces of the dehydrated dentin beams resulting from flexure. The top of the beam is the tensile surface: (a) fracture surface from a beam that failed at a low stress rate (0.01 MPa/s) and (b) fracture surface from a beam that failed at a high stress rate (100 MPa/s).

the elastic modulus resulting from experimental studies of dentin ranges from less than 10 GPa to nearly 30 GPa. Thus, the range in flexure modulus obtained for the bovine dentin agrees overall with results from previous studies of dentin. The rise in flexure modulus with stress rate emphasizes the significance of time-dependent deformation on the elastic response of dentin.

In an evaluation of the viscoelastic behavior of dentin, Jantarat et al.[8] found that the elastic modulus of hydrated human root dentin increased only 2–3 GPa (from ~14 to 17 GPa) with increase in strain rate from $1 \times 10^{-4}$ to 5 $s^{-1}$. However, the rise in flexure modulus for the bovine dentin reached an average increase of 4.75 GPa per order magnitude increase in strain rate. Results of the present study suggest that hydrated dentin exhibits a much larger strain rate sensitivity than that found by Jantarat. Although there is a difference in the source of dentin between the two studies, the primary difference is the mode of loading; Jantarat used uniaxial compression. Differences in the mode of loading are relevant because of potential differences in contribution of the collagen fibrils and hydraulic phenomena.[5,7,9] Interestingly, Fan and Rho[25] examined the time-dependent response of human cortical bone using nanoindentation with several different load histories over strain rates from $\sim 1 \times 10^{-4}$ to $1 \times 10^{-2}$ $s^{-1}$. In examinations using a single monotonic indentation load history, the elastic modulus was proportional to the strain rate raised to the 0.1 power. Using their power law model over the range of strain rates used in evaluating the dentin, the apparent modulus for bone would range from ~12 GPa to nearly 31 GPa, an increase of ~4.6 GPa per order magnitude increase in strain rate. These results are very consistent with those obtained for the bovine dentin herein. Differences in the degree of rate-dependence in the elastic response of dentin suggest that the mode of loading and corresponding stress state may be relevant to the time-dependent response of dentin. Uniform compression could exhibit a lower degree of time dependence due to diminished contribution of collagen fibrils in compression relative to that in tension, or due to differences in water movement. These comments are speculative and further study is required to identify the specific mechanisms responsible for the rate-dependent mechanical behavior.

Previous studies on the strain rate-dependent response of dentin have focused on the elastic behavior but have not identified the changes in strength or energy to fracture. The flexural strength of the hydrated dentin increased from ~100 MPa ($(\dot{\sigma})$ = 0.01 MPa/s) to 250 MPa ($(\dot{\sigma})$ = 100 MPa/s) over the range in stress rate. In an evaluation of bovine incisors,[26] the ultimate tensile strength (UTS) of dentin reportedly ranged from 90 to 130 MPa. Thus, it appears that results from flexure loading at the lower stress rates are consistent with previous results reported for the UTS of bovine dentin. Many recent studies of dentin have evaluated the importance of tubule orientation and tubule density to the tensile strength [ for e.g. Ref. 27], and in comparison, there has been limited emphasis on the rate dependence of strength. However, the influence of load rate on the strength of cortical bone has been of interest for many years. In early notable studies of bone,[28–30] the ultimate strength was found to increase from ~100–300 MPa over strain rates increasing from $1 \times 10^{-4}$ to 100 $s^{-1}$. These results agree with the overall range in flexure strength for the bovine dentin and confirm that there is a distinct reduction in strength of dentin with decrease in stress rate. The rate-dependent reduction in strength of bone has been attributed to the rate of damage accumulation and quantitatively described using the 'cumulative damage model'.[31,32] Results from flexure tests are not sufficient to identify the specific mechanisms that enable damage coalescence in dentin at low load/stress rates.

The change in flexure strength and energy to fracture of dentin with stress rate indicates that restorative treatments and/or appliances can contribute to the apparent damage tolerance of dentin. Treatments that cause development of static (time invariant) stress (e.g. residual stress due to polymerization shrinkage of resin composites, orthodontic appliances) would appear to cause a significant reduction in energy to fracture [Fig. 6(c)]. In fact, fracture surfaces of the hydrated dentin beams suggest that there is a transition from brittle behavior at low stress rates [Fig. 7(a)] to something more consistent with ductile behavior at high stress rates [Fig. 7(b)]. Although it is unlikely that static (time-invariant) stresses will foster bulk fracture of the tooth, they may facilitate development of damage and/or flaws that undergo cyclic extension and eventually sacrifice structural integrity of the tooth. A recent evaluation of fatigue crack growth in dentin considered the contribution from time-invariant stresses on the rate of cyclic crack growth according to different stress ratios.[33] Results of that investigation distinguished that an increase in the cyclic stress ratio from 0.1 to 0.5 (which promotes an increase in the magnitude of static stress) resulted in a reduction in stress intensity necessary for initiation of a fatigue crack and a significant increase in the cyclic crack growth rate. Therefore, restorative treatments that introduce a monotonic stress within the tooth may cause a reduction in the damage tolerance of the dentin, which facilitates the initiation and rapid growth of flaws that result in tooth fracture.

A change in the mechanical behavior of dentin attributed to level of hydration is a relevant clinical issue. A study of age and moisture[34] showed that teeth over 50-years-old contained less water than young teeth (10–20 years of age). During aging, it is common for the dentinal tubules to become filled with mineral crystals, thereby lowering the water content of dentin through reduction in the internal delivery of fluids. Occlusion of the tubules appears to be responsible for decreased fatigue strength[35] and is consistent with the decrease in strength and energy to fracture [Fig. 6(a,c)] with dehydration in the present study. Perhaps also relevant, Xerostomia (dry mouth) is common in seniors[36,37] and could cause further reduction in external tooth hydration through reduction in saliva. A comparison of fracture surfaces from the hydrated and dehydrated dentin showed that hydration changed the intrinsic behavior of the tissue as noted in Figure 7(b), a shear lip regularly developed on the compressive side of the hydrated specimens loaded at higher stress rates. A compression shear lip has also been identified on the fracture surface of specimens from fully hydrated young human dentin where the tubules were oriented perpendicular to the length of the beam.[35] The shear lip developed as a result of the mechanical anisotropy and the preference for fracture perpendicular to the tubules, the orientation with lowest fracture toughness.[38] Absence of a shear lip on the bovine dentin specimens at lower stress rates suggests that the mechanisms promoting mechanical anisotropy were suppressed or that fracture proceeded along an alternative path due to the population of flaws that developed with time. It could also suggest that the dentin was "embrittled". But the term "brittle" does not appear to be completely appropriate if the flexure responses are evaluated in terms of the strain to fracture (Fig. 9). While the hydrated specimens exhibited low strength at the low stress rates, the strain to fracture was 0.008 m/m and nearly independent of the stress rate (Fig. 9). Interestingly, the average strain to fracture of the dehydrated dentin was approximately half that of the hydrated tissue (0.004 m/m) and nearly independent of strain rate as well.

The unique trends in mechanical properties of the hydrated and dehydrated dentin with stress rate have not been identified previously. Nevertheless, the influences of hydration on the stiffness, strength, and fracture properties of dentin have recently been examined. Nalla et al.[39] evaluated the role of hydration on the fracture properties of elephant tusk dentin where dehydration was achieved using different "water-free" polar solvents (i.e. acetone, ethanol, and methanol). In that study, it was found that dehydration with

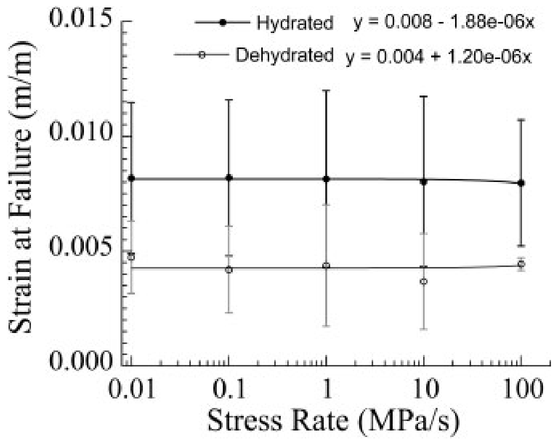


**Figure 9.** The influence of hydration and stress rate on the strain to fracture of the dentin beams.

polar solvents caused an increase in stiffness of dentin and also promoted an increase in both the intrinsic and extrinsic components of fracture toughness. The changes in properties were completely reversible with hydration and could be recovered again with redehydration. They rationalized that dehydration with the polar solvents enabled an increase in the number of hydrogen bonds between adjacent collagen peptide chains, a theory also proposed by Pashley et al.[40] in the evaluation of hydration effects on demineralized dentin. An increase in the intermolecular hydrogen bonding was expected to cause an increase in the strength and stiffness of the fibril network, thus providing increased resistance to cracking and additional bridging forces that reduced the energy available for crack extension. The increase in direct collagen-collagen bonding and potential contraction of the fibrils, have also been suggested to invoke compressive residual stresses on the mineralized tissue, further increasing the resulting stiffness.[40] Indeed, results for the bovine dentin in Figure 6 show that there is a significantly larger strength, elastic modulus and energy to fracture of the dehydrated dentin at low stress rates. However, with increase in stress rate there is a distinct reduction in the strength and energy to fracture of the dehydrated bovine dentin [Fig. 6(a,c)], respectively, while there is marginal increase in elastic modulus. If these results are attributed to the additional intermolecular hydrogen bonds and increased participation of collagen fibrils, they also indicate that these bonds are far more rate sensitive than the weaker hydrogen bonds (in the presence of water) in hydrated dentin and that they adversely affect the strain to fracture (Fig. 9). The viscoelasticity of the collagen fibrils, which may be associated with movement of water through the tissue with application of mechanical loads, is also likely relevant to the increase in strength and toughness of the hydrated dentin with stress rate. Based on these observations, it appears necessary to also recognize the potential importance of stress rate in future studies on the properties of the collagen fibrils and their contribution to the bulk properties of dentin.

The slow crack growth exponent (n) represents the material's sensitivity to rate of loading and materials with a high n are less sensitive to stress or strain rate. Results obtained for the glass (Table II) provided a slow crack growth exponent of 199, denoting a response that is essentially insensitive to stress rate. The hydrated dentin exhibited an $n$ of 9, indicating that the flexure behavior of hydrated dentin was far more time-dependent than glass and that the strength increases with stress rate. In contrast, the n for dehydrated dentin was found to be $-21$, indicating that dehydration reduced the rate dependence and that there is a decrease in strength with stress rate. Nevertheless, the exponent for dehydrated dentin suggests that the tissue remains partially time-dependent (e.g. in comparison to glass), which again is very likely attributed to properties of the collagen fibrils.

As is true with most experimental investigations, there are recognized limitations to this investigation. The dynamic fatigue experiments were conducted at room temperature, whereas loading within the oral cavity occurs at 37°C. Previous investigations comparing the mechanical behavior of dentin over this temperature range suggest that although a decrease in the elastic modulus would be expected with increase in temperature,[41] the change in strength and energy to fracture would be small.[42] In addition to temperature concerns, the experiments were conducted in a calcium-buffered saline bath. Recent studies using nanoindentation[43] have shown that demineralization of dentin occurs in this medium within a 3-week period, which, therefore, could have influenced the dynamic fatigue responses. Demineralization would promote an increase in the compliance and consequent reduction in the elastic modulus. Nevertheless, the specimens were evaluated within 3 weeks of extraction, and the similarity in elastic modulus between bovine and human dentin suggests that potential changes due to demineralization were small. Despite these concerns, this study has provided additional fundamental knowledge on the mechanical behavior of dentin. Most importantly, the results indicate that the strength and energy to fracture of hydrated dentin is lowest under conditions that promote development of static stresses. Thus, the results provide further insight on the potential contribution from restorative practices on restored tooth failures through changes in the mechanical behavior of tooth tissues that arise with oral factors contributing to stress rate and hydration.

## CONCLUSIONS

An experimental evaluation of the dynamic fatigue response of dentin was conducted. Rectangular beams of dentin were prepared from maxillary molars of mature bovine and subjected to four-point flexure with stress rate ranging from 0.01 to 100 MPa/s. The evaluation was conducted with dentin beams in the fully hydrated condition and after complete dehydration. Based on results from this study, the following conclusions were drawn as follows:

a. The flexure modulus of the hydrated dentin increased from an average of 16–35 GPa with an increase in stress rate from 0.01 to 100 MPa/s, respectively. The flexure modulus of the dehydrated dentin increased from 22 to 28 GPa over the same range in stress rate.
b. The flexure strength and energy to fracture of the hydrated dentin increased significantly with increasing stress rate (($\dot{\sigma}$)). The flexural strength increased from 100 MPa (($\dot{\sigma}$) = 0.01 MPa/s) to 250 MPa (($\dot{\sigma}$) = 100 MPa/s). The fracture energy increased from 0.40 MPa (($\dot{\sigma}$) = 0.01 MPa/s) to 1.4 MPa (($\dot{\sigma}$) = 100 MPa/s).
c. In contrast to hydrated dentin the flexure strength and energy to fracture of the dehydrated dentin decreased with increasing stress rate. The strength decreased from 170 MPa (($\dot{\sigma}$) = 0.01 MPa/s) to 100 MPa (($\dot{\sigma}$) = 100 MPa/s). Similarly, the energy to fracture decreased from 0.7 to 0.2 MPa over the aforementioned range in stress rate.
d. Hydrated dentin beams that were loaded at a high stress rate exhibited a compression shear lip on the fracture surface. Neither the hydrated beams loaded at low stress rates nor the dehydrated beams exhibited a compression shear lip.

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