Measurement of biomechanical behavior of dentin hard tissue in response to unbound water loss using stereo-digital image correlation

Zhenning Chen¹,³ and Xinxing Shao²,³

¹ College of Aerospace Engineering, Nanjing University of Aeronautics & Astronautics, Nanjing 210016, People’s Republic of China
² Department of Engineering Mechanics, Southeast University, Nanjing 210096, People’s Republic of China
³ Authors to whom any correspondence should be addressed.

E-mail: zhenning.chen@nuaa.edu.cn and xinxing.shao@seu.edu.cn

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Abstract
Understanding the biomechanical behavior of dentin hard tissue with fluid-filled dentin tubules and hydrated matrices is essential for studying this functionally graded biological composite. The stereo-digital image correlation technique with an adaptive high-magnification field of view (FOV) for fully hydrated biological tissue measurement was investigated. The adaptive magnification is controlled by the length of extension tubes. To determine both the unbound water loss induced and load-induced three-dimensional (3D) deformation of dentin hard tissue from a fully hydrated state to a non-hydrated condition, samples of dentin blocks and half teeth in sagittal sections were studied for a period of 2 h in situ over varied speckle patterns. The effects of speckles on water evaporation, camera pre-heating, and measurement accuracy in the wet, curved and long-term measurement were analyzed. The elastic modulus and Poisson’s ratio of both dentin and pulp in response to unbound water evaporation were measured. With the unbound water loss, the mean values of the elastic modulus generally increased from ∼8 GPa to ∼10 GPa in pulp region and from ∼10 GPa to ∼12 GPa in dentin region. The mean values of the Poisson’s ratio increased both in pulp and in dentin. Poisson’s ratio in the dentin regions (∼0.3) were generally smaller than those in the pulp regions (even can reach 0.6), irrespective of the partial dehydration time. Further analysis of the full-field deformation results provided insight into the unbound water-induced regional deformations and mechanical changes in human dentin. It’s found that the unbound water loss induced deformations were more prominent when compared to load induced deformations.

1. Introduction

Deformation measurement of biological composites under the influence of external forces is crucial in understanding its biomechanical properties and approximate them with artificial materials. Human dentin hard tissue is a porous, functionally graded, biological composite which forms the bulk of the crown and the root of the teeth. It comprises 70% of carbonated apatite crystals, and 20% of collagen and proteins, with approximately 10% of water by weight [1]. Water circulating in the fluid-filled dentin tubules and the hydrated matrices of dentin consists of bound water (∼70%) and unbound or free water (∼30%) [2, 3]. In dentistry, unbound water loss in dentin is inevitable during endodontic treatment, and some studies have attributed the increase in fracture susceptibility of endodontically treated teeth to a decrease in moisture content of dentin. However, due to complex tooth structures and biological compositions, mechanical properties vary conspicuously with respect to spatial distributions. Thus, a investigation of the full-field, three-dimensional (3D), high-resolution experimental technique to measure complex biological composites under physiologically realistic loads and conditions is important. It is also critical to the development and application of photo-biomechanics.

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Photo-biomechanics is a discipline of science that utilizes optical techniques to study the behavior of biological structures under function or external forces [4]. Optics-based experimental techniques are applied for measuring full-field physical quantities, such as stress, profile, displacement and strain. In dentistry, two-dimensional (2D) digital image correlation (2D-DIC) is generally utilized to measure in-plane deformation of flat dental specimens [5]. However, 2D-DIC has its limitations, when it comes to non-flat samples and out-of-plane deformation induced in-plane errors. 3D Electronic speckle pattern interferometry (3D-ESPI) is used to assess the impact of different restorative materials on the deformation of teeth [6]. Digital moiré interferometry (DMI) was applied to decipher the dentin tissue biomechanics by fabricating gratings with ultra-high quality on an extremely flat surface [7]. Brozović J used digital holographic interferometry (DHI) to determine the properties of axially loaded dental implant–abutment assemblies [8]. Asundi and Kishen utilized photoelasticity to evaluate stress distribution, from the root surface to the supporting bone under clinical conditions [9]. For long-term monitoring of wet, curved dentin samples, these laser-interferometry based techniques possess inherent challenges due to their environmental sensitivity. In general, very few studies have worked on the measurement of 3D deformation states (e.g., in-plane and out-of-plane displacements, strains) of complex biological composites, such as dentin structures, from a fully hydrated condition to a non-hydrated state. In previous studies, we adopted the stereo-digital image correlation (stereo-DIC) technique to characterize long-term deformations of wet, curved dentin during unbound water loss [10]. However, the elastic modulus and Poisson’s ratio of dentin varied as unbound water loss weren’t measured and the system errors weren’t analyzed [10, 11], which is an important task to understand the affection of unbound water loss and mechanical loading in endodontically treated teeth.

With cold light illumination and two synchronized cameras to monitor flat or curved objects, stereo-DIC turns to be an advanced optics-based experimental measurement technique, with strong 3D variables measuring abilities [12]. In general, the speckle will be prefabricated on the specimen surface to enhance the intensity variance before stereo-DIC measurement. The randomly distributed speckle is essential for obtaining accurate and efficient deformations [13–15]. Previous fabrications of the speckle patterns on biological materials have been reported in the measurement of deformations of relatively rigid tissues, such as the cartilage and the hoof horn [5, 16], or soft stiff tissues such as the arteries [17] and skins [18], or very soft tissues like the liver tissue [19]. Usually, the speckle pattern used for biological tissues was generated by spraying either quick-drying paint, toner powders, or silicon carbide particles, or using the natural texture exhibited on the surface like porcine liver tissue [19]. In Berfield’s study, a fluorescent particle solution was spin–cast onto the samples to achieve real-time measurements of nanoscale deformations [20]. Ning further applied both, fluorescent microsphere binding and ethidium bromide nuclear staining to generate high- contact random patterns on mouse carotid arteries [17]. Considering the pattern detachment, Thompson used powder ink mixed with a gelatinous substance and applied it on a small flat section of a sheep bone callus [21]. Lionello used saline solution containing 1% methylene blue, with small white paint and applied it on unshaped porcine collateral ligaments to obtain nearly 10% nominal elongation [22]. Libertiaux used talcum powder to prevent light reflections of brain tissue [23]. Besides the probability of paint film delamination and crumbling, speckle fabricated on wet, curved dentin may block water evaporation in dentinal tubules. This will further alter the biomechanical behavior of the tissue in response to unbound water loss. However, few studies have investigated the effects of speckles on water evaporation during the full-field biomechanical behavior of dentin hard tissue, from a fully hydrated condition to a non-hydrated state, especially for the effects on water evaporation.

In this study, an adaptive high–magnification stereo-DIC measurements were utilized to examine the full-field biomechanical behavior of dentin hard tissue. The effects of speckles on water evaporation, camera pre-heating, and measurement accuracy in this wet, curved and long-term measurement were analyzed. The mechanical parameters during unbound water loss were measured using block slices samples. Full-field experiments under mechanical loadings and unbound water losses were carried out in the sagittal plane of the half teeth and the sectioned slice.

2. Stereo-DIC technique for fully hydrated biological tissue measurement

2.1. 3D deformation measurement using stereo-DIC

Based on the stereovision theory and the 2D DIC approach, stereo-DIC has proven to be an advanced and powerful optics-based experimental technique with strong 3D variables measuring abilities [24]. To investigate the biomechanical behavior of dentin hard tissue in response to unbound water loss and mechanical loading, an adaptive high–magnification stereo-DIC imaging system was utilized to examine the full-field deformation of dentin hard tissue under mechanical loadings and unbound water losses. Figure 1 shows the stereo-DIC setup and the principle of stereovision. In general, two synchronized cameras are used in the stereo-DIC imaging system. To achieve an adaptive, high–magnification field of view (FOV), 2 high-resolution cameras (CMOS,
spatial resolution 2048 × 2048 pixels, 80 fps, UI-3370CP-M-GL, IDS, Obersulm, Germany), a pair of TV lenses (F1.8–14, 50 mm, C-mount, Pentax, Japan), and extension tubes (C-Mount, Edmund Optics, Barrington, Illinois) are mounted as shown in figure 1(a). The length of extension tubes is used to control the magnification of imaging. This configuration allows FOV range from 10 × 10 mm² to 20 × 20 mm², which is adequate for the experiments described in section 3. The VIC-3D software [25] is used to analyze the experimental images and obtain the full-field deformation. During the camera calibration, the coordinates of principle points are fixed on the center of the image.

2.2. Effects of speckles on water evaporation
Stereo-DIC matches gray intensity information of the specimen surface with the prefabricated speckle between the reference and the target image. Hence, the randomly distributed speckles are essential for obtaining accurate and efficient deformations [26]. In our experiment, human dentin hard tissue is a porous composite filled with water, so the fabricated speckles should adhere to the hard tissue, irrespective of water evaporation. In the speckle fabrication procedure, white matte paint was not sprayed as usual because of the partly milky white color of the dentin surface. Instead, black matte dots were uniformly sprayed onto the milky white dentin directly. A microscopic investigation of the speckles on dentin slice was performed. Firstly, the dentin slice with sprayed black speckle was put on a glass. Then, the contact surface between the dentin slice and glass was focused and imaged under a light microscope. Through the microscope, free water in the dentin slice evaporated from dentinal tubules and condense onto the contact surface. Thus, free water evaporation can be qualitatively imaged by the microscope. Figure 2 shows the microscopy images recorded every 5 s. The blurred black stands for speckle, as marked in figure 2(a). The upper row (figures 2(a)–(d)) shows water evaporation near the canal area of dentin, and the lower row (figures 2(e)–(h)) shows the water loss near the dentin-enamel-junction (DEJ) area of dentin.
Dentin forms the major bulk of the human tooth. It constitutes thousands of microscopic channels, called dentinal tubules that traverse almost the entire bulk of dentin. The unbound water that is composed of approximately 30% of all the water in dentin is found to occupy the dentinal tubules predominantly and the fine microbranches of the dentinal tubules (75.2%) over the mineralized dentin matrix [2, 3]. In this investigation, speckle particles on the test dentin slice was about 100 μm, while the lumens of the dentinal tubules was only several microns (vary in diameter: from 0.5–0.9 μm DEJ to 2–4 μm through the direction toward pulp, 4.3–1.7 μm from coronal to apical, in density: 20000–45000/mm² from DEJ to pulp, 72%–13% from coronal to apical).

On comparing the upper row (figures 2(a)–(d)) and the lower one (figures 2(e)–(h)), it is seen that faster water evaporation occurs in dentinal areas with larger and denser tubules. As water evaporates from the dentinal tubules, the speckles, especially the large particles, will obstruct water evaporation directly from the end of the dentinal tubule (see the largest speckle in figures 2(a)–(d)). However, dentinal tubules are connected in such a way that water can evaporate even where there are speckles (see the small speckles in figure 2). In other words, sprayed speckles will not completely obstruct full-field water evaporation but will decelerate it. The sprayed black speckles can adhere to the dentin surface well. Thus, for similar stereo-DIC measurements of fully hydrated biological tissues, sprayed black matte paint can be used.

2.3. Accuracy verification
During the stereo-DIC measurement of the fully hydrated biological tissue, the heat balance of both the cameras should be considered, in order to achieve accurate results. Before conducting the experiments, a rigid plate (the thermal expansion of the plate is negligible) with printed digital speckles was monitored by the camera, with an activation of about 100 min. This test was repeated five times. Thereafter, in-plane strain components $\varepsilon_{xx}$ and shear strain $\varepsilon_{xy}$ were calculated with stereo-DIC, which was activated to 100 min. Statistical results with time changes are shown in figures 3(a)–(c). Regions R0–R4 are shown in figure 3(a). From figures 3(a)–(c), it can be seen that the stereo-DIC system will cause more than 200 με strain error after about 1 h of imaging. Hereafter, this strain error will reach equilibrium. Such a large strain error is unacceptable and should be eliminated [27].

In order to ensure that the measurement accuracy can meet the requirement, the two cameras are preheated before measurements. Figure 3(d) shows the statistical strain errors of the 5 groups after 1 h of preheating in the stereo-DIC system. Mean bias errors of each group were lower than 25 με and their standard deviation error were lower than 40 με, except for group 3. After preheating, the measurement accuracy is enough to conduct the dentin experiment.
3. Experiments

3.1. Sample preparation
The Research Ethics Board of the University of Toronto approved the collection of teeth for this study (protocol #5975). Seven freshly extracted human mandibular premolars with a single straight root canal were collected from young patients (11 ≤ age ≤ 30) and stored in deionized water at 4 °C. Only non-carious teeth without visible cracks, fractures or restorations were selected. Three teeth were sectioned into 6 block slices (about 9 mm × 4 mm × 0.7 mm) under a water coolant. The block slice was sectioned from the rectangular area, as shown in figure 4. The remaining four teeth were accessed with a fissure bur, and instrumented using #10 K-File, followed by ProTaper Gold S1-S2-F1-F2-F3 rotatory instruments. Patency was maintained, and the irrigation was done with a 2.5% sodium hypochlorite (NaOCl) solution. A diamond disc was used to section these teeth into 8 halves in a buccal-lingual direction, under the exposure of a water coolant. In order to enhance the adhesion of speckles for stereo-DIC, the sectioned blocks and the eight halves were slightly cross grounded with wet emery paper of grit size 800. 5 block slices were used for long-term experimental measurement of the elastic modulus and Poisson’s ratio in different water loss statuses. 8 halves were used for half-teeth mechanical loading experiments. All specimens were placed in an ultrasonic bath and cleaned using a 2.5% NaOCl solution for 3 min, followed by a 17% ethylene diamine tetra acetic acid solution for 1 min, and finally, by deionized water for 3 min to remove the smear layer from the root canal walls. Then, all specimens were stored in deionized water at room temperature for at least 24 h to allow for rehydration. All specimens were tested within 4 weeks, following storage. About 5 min before corresponding stereo-DIC measurements, each specimen was taken out of deionized water and sprayed with black speckles in the fume hood, as described in section 2.2. Thereafter, the specimens were rehydrated in deionized water for a while. The stereo-DIC measurements were then performed. The steps taken during the specimen preparation followed that of our previous experiments [10].

3.2. Elastic modulus and Poisson’s ratio
Measuring the elastic modulus and Poisson’s ratio of dentin is important, as the application of adhesive restorative materials and biomechanical simulations of teeth depends largely on the distribution of these mechanical properties. It is anticipated that as in vivo dentin is wet and unbound water-loss of dentin is inevitable during endodontic treatment, testing the elastic modulus and Poisson’s ratio of dentin from a fully hydrated state to a non-hydrated state may help in understanding the fracture susceptibility of endodontically treated teeth. The setup used in this experiment is shown in figure 1(a). For long-term monitoring, the setup was fixed on the air suspension table. Sizes of 5 block slices were carefully measured and sprayed with speckles following section 2.2. Two ends of each specimen were decorated with polyvinyl siloxane to avoid jig abrasion when mounted on a customized loading jig, as shown in figure 5(b). Compressive loads ranging from 0 to 40 N with a 5 N load increase were sequentially applied along the direction, as shown in figure 5(a). To measure the elastic modulus and Poisson’s ratio of dentin induced by both unbound water loss and dentin structure, the same loading procedures were performed after 30, 60, 90, and 120 min of partial dehydration. The specimens were imaged using the stereo-DIC setup after each load application. 3D full-field analysis of the acquired images...
was performed to determine the full-field deformations. Results of both dentin region (circled with 1 in figure 5(a)) and pulp region (circled with 2 in figure 5(a)) were compared.

### 3.3. Half-teeth mechanical loading

Deformation response to mechanical loading and unbound water loss was affected by both tooth structure and composition. It is anticipated that as *in vivo* dentin was wet and unbound water-loss of dentin was inevitable during endodontic treatment, testing of the half-teeth from a fully hydrated state to a non-hydrated state may help in understanding the fracture susceptibility of endodontically treated teeth. This experiment also used the same setup as shown in figure 1(a). Before testing, the samples were sprayed with speckles following section 2.2. Each specimen was slightly sectioned to obtain a flat loading end. When mounted on a customized loading jig, the two ends of each specimen were carefully decorated with polyvinyl siloxane to avoid jig abrasion, as well as to simulate the supporting of the alveolar bone, as shown in figure 6(b). Compressive loads ranging from 0 to 100 N with a 10 N load increase were sequentially applied along the direction shown in figure 6(a). The same loading procedures were performed after 30, 60, 90, and 120 min of partial dehydration. The specimens were imaged using the stereo-DIC setup after each load application. 3D full-field analysis of the acquired images was performed to determine the full-field deformations.

### 4. Results

#### 4.1. Elastic modulus and Poisson’s ratio

After the full-field image analysis described in section 2.1, strains in the pulp and dentin regions of the block slices (see in figure 5) were extracted to obtain the corresponding mechanical properties. As seen in figure 7(a) and figure 7(c), the absolute strain $\varepsilon_{xx}$ and $\varepsilon_{yy}$ values increased after 0, 30, 60, 90, and 120 min of partial dehydration at the same load level. This is due to the residual strain induced by unbound water loss. Especially in the initial 30 min of partial dehydration, the unbound water loss induced deformations were more prominent when compared to mechanical load-induced deformations. Both the elastic modulus $E$ and Poisson’s ratio $\nu$ were calculated from the stress-strain curves shown in figures 7(a) and (c). The equations are described below.
\[ \sigma = \frac{F}{A} \]  
\[ E = \frac{\sigma}{\varepsilon_{xx}} = \text{slope}_{xx} \]  
\[ \nu = -\frac{\varepsilon_{yy}}{\varepsilon_{xx}} = -\frac{\text{slope}_{yy}}{\text{slope}_{xx}} \]  

where \( F \) is the compressive load, \( A \) is the area of cross section perpendicular to the loading direction (x direction), \( \varepsilon_{xx} \) is the calculated local strain in the x direction, \( \varepsilon_{yy} \) is the calculated local strain in the y direction, the locations of extracted local strain are shown in figure 5 with number 1 and 2, \( \text{slope}_{xx} \) and \( \text{slope}_{yy} \) are the slopes of linear fitting stress-strain curves \( \varepsilon_{xx} - \sigma \) and \( \varepsilon_{yy} - \sigma \), respectively.

Using equation (2), the elastic modulus was calculated, and the statistical results of the elastic modulus are shown in figure 7(b). With the unbound water loss, the mean values of the elastic modulus generally increased from \( \sim 8 \) GPa to \( \sim 10 \) GPa in pulp region and from \( \sim 10 \) GPa to \( \sim 12 \) GPa in dentin region. So fully hydrated sample had smaller elastic modulus. Mean values in the pulp regions were smaller than those in the dentin regions at the same partial dehydration time. Thus, pulp region turned to have the smallest elastic modulus. The elastic modulus in the pulp region in the fully hydrated (wet) state was statistically lower than that in peripheral dentin region, in the unbound water loss (partially dehydration) state.

Using equation (3), the Poisson’s ratio was calculated, and the statistical results of Poisson’s ratio are shown in figure 7(d). With the unbound water loss, the mean values of the Poisson’s ratio increased both in pulp and in dentin. Poisson’s ratio in the dentin regions (\( \sim 0.3 \)) were generally smaller than those in the pulp regions, irrespective of the partial dehydration time. Distribution of the Poisson’s ratio in pulp regions showed larger fluctuations than in dentin.

4.2. Half-teeth mechanical loading

Figure 8 shows a typical loading-deformation curve. Here, deformation \( U \) stands for the total displacement of the loading end along the loading direction given in figure 6. The total displacement is induced by both partial dehydration and subsequent loads, i.e., deformation \( U \) is a sum of partial dehydration-induced \( U \) and subsequent load-induced \( U \). In the first part of figure 8 (part I, loading index from 0 to 10), deformation \( U \) of the wet/fully hydrated sample in response to compressive loads ranging from 0 to 100 N with a 10 N load increase were plotted. Deformation \( U \) in response to the same loading procedures after 30, 60, 90, and 120 min of partial dehydration.
dehydration were also plotted in figure 8 (part II, III, IV, and V, loading index from 11 to 21, 22 to 33, 34 to 44, and 45 to 55, respectively). According to figure 7(b) in section 4.1, fully hydrated sample has smaller elastic modulus than sample after partial dehydration, so deformation U of wet sample in response to mechanical loads should be bigger than that of partially dehydrated sample. However, as shown in figure 8, deformation U of partially dehydrated sample is generally bigger than deformation U of wet sample. Due to the water loss, partial dehydration-induced part of deformation U (loading index 11, 22, 33, 44) is bigger than subsequent load-induced part (<0.5 mm), except at the fully hydrated state (part I). Thus, partial dehydration-induced displacement was more prominent when compared to subsequent load-induced displacement.

In the full-field image analysis, both partial dehydration-induced and subsequent load-induced deformations were investigated. Strain distributions of half-teeth induced by both 100 N mechanical loading and 2 h unbound water loss, showing the differences in macro structural distribution and mechanical properties, are given in figure 9. The upper row stands for the deformation (strain $\varepsilon_{xx}$, $\varepsilon_{yy}$, and $\varepsilon_{xy}$) under 100 N mechanical loading (loading index 10 of figure 8). An intermediate deformation (strain $\varepsilon_{xx}$, $\varepsilon_{yy}$ and $\varepsilon_{xy}$) is shown in the middle row where deformation is induced only by partial water loss (loading index 44 of figure 8). The lower row stands for deformation under the influence of a load of 100 N after 2 h partial dehydration (loading index 55 of figure 8). Strain concentrations in enamel and the outer dentin, near the roots, were detected and marked with either circles (tensile strain) or triangles (compressive strain). The deformation comparison under three different loads and dehydration conditions showed an overall increase in compressive strain $\varepsilon_{xx}$ in the dentin area, due to both partial dehydration and compressive loads (see figure 9(a), figure 9(d), figure 9(g)). Partial dehydration-induced tensile strain $\varepsilon_{xx}$ was detected in enamel and in the regions circled in figure 8(d). However, the tensile strains in the circular regions had smaller values when the compressive loads were applied in figure 9(g). Partial dehydration-induced concentrated residual strain $\varepsilon_{yy}$ was also detected in regions localized near the apical dentin and the DEJ. Moreover, partial dehydration-induced deformations (strain $\varepsilon_{xx}$, $\varepsilon_{yy}$ and $\varepsilon_{xy}$) were more prominent when compared to load-induced deformations (see each column of figure 9).

5. Discussions

As the major bulk of the human tooth, dentin consists of thousands of fluid-filled dentinal tubules, decreasing from the coronal to the apical aspect and from the pulp to the peripheral aspect of the tooth. Lumens of dentinal tubules vary in diameter: from 0.5–0.9 μm DEJ to 2–4 μm through the direction toward pulp, 4.3–1.7 μm from coronal to apical, in density: 20000–45000/mm² from DEJ to pulp, 72%–13% from coronal to apical. Due to the spatial variations of the tooth structure and the composition, the elastic modulus varies conspicuously. Previous studies have shown that the elastic modulus of enamel is 40–80 GPa, while it is around 30 GPa in peritubular dentin and 16–21 GPa in inter-tubular dentin. It may be even lower (3–19 GPa) in regions as close as 500 μm from the pulp [28, 29]. Chan et al. found that enamel had an elastic modulus of ~95 GPa, whereas dentin had an elastic modulus of ~19 GPa from nano-indentation tests [30]. Angker et al. found that the elastic modulus of the area near the pulp region was statistically lower than those of dentin in the middle and near the DEJ [29]. Kinney et al. used the atomic-force microscope indentation technique to determine the elastic modulus of inter-tubular dentine. He found that there was a rapid decrease in elastic modulus in the first 24 h of rehydration [31]. The stereo-DIC investigations showed that the elastic modulus in the fully hydrated (wet) state was statistically lower than that in the unbound water loss (partially dehydration) state (in the pulp region from ~8 GPa to ~10 GPa, in
peripheral dentin region from ∼10 GPa to ∼12 GPa), i.e. the elastic modulus in wet pulp was statistically lower than that in unbound water loss pulp, and the elastic modulus in wet dentin was statistically lower than that in unbound water loss dentin. Since pulp region has bigger lumens accommodated with more free water, a conclusion that dentin hard tissue with more free water has the lower elastic modulus can be obtained.

Kinney et al. found that differences of the elastic properties changed by transparency (ages) were not significant [32]. Zhang also found that the shrinkage strains in both of the dentin and enamel tissues were isotropic and there was no significant difference in the dimensional changes between tissues from the young (18 ≤ ages ≤ 30) and old (ages ≥ 50) patients [33]. Teeth used in the stereo-DIC experiments were extracted due to the orthodontic treatment. Most patients getting orthodontic treatment were usually between 11 and 15 years old but some were between 20 and 30. Thus, changes of the age-related elastic modulus were not considered. However, the mechanical properties of dentine, such as microhardness, roughness, elastic modulus, flexural and fatigue strength, can be influenced by treatment with NaOCl [34]. The studies included in Pascon’s review showed reductions in the elastic modulus and flexural strength of dentin after irrigation of the root canals with 2.5%, 3%, 5%, 5.25%, and 9% NaOCl within from 24 min to 2 h [34]. From the clinical point of view, it would be prudent to select a suitable NaOCl concentration, which had minimal effects on the mechanical properties of the tooth while achieving the desired debridement effect. In our experiments, all specimens were cleaned using a 2.5% NaOCl solution for 3 min irrigation, which would have inevitable but less deteriorating effect on the mechanical properties.

Previous studies have shown that the Poisson’s ratio of the inter-tubular matrix varies from 0.1 to 0.4. There was considerable anisotropy in the Poisson’s ratio in mineralized wet dentin (ν21 = 0.45, ν31 = 0.29). This anisotropy ceased to exist with drying in air (ν21 = ν31 = 0.29) [35]. The stereo-DIC investigations showed that the mean value of Poisson’s ratio in pulp was bigger than those in the dentin region (ν ≈ 0.3). Also, the values, both in pulp and dentin regions, increased as the unbound water loss increased.

Samples investigated in the half-teeth mechanical loading was sectioned included the enamel tissues, DEJ and cementum tissues [30, 36]. Cementum under wet condition was proved to have smaller elastic modulus
than in dry condition [36]. Herein the cementum tissues were at the edge of our samples, and stereo-DIC technique has limited ability to compute the edges. Enamel and DEJ were important tissues that play an important role in distributing the load and they were extremely affected by the dehydration. Enamel was found to have an elastic modulus of $\sim 95$ GPa, whereas dentin had an elastic modulus of $\sim 19$ GPa, and a sharp change in mechanical properties was observed across DEJ to have the low delamination probability due to collagen [30]. In our previous work [10], distributions of the water-loss induced deformations in dentin, enamel, and DEJ were investigated particularly. Here, both partial dehydration induced and subsequent load-induced deformations were studied. In the further full-field image analysis of both partial dehydration induced and subsequent load-induced deformations, the differences in tooth macro-structural distribution indicated more prominent deformation than the differences in the obtained elastic modulus and Poisson’s ratio. Enamel regions were found to have relative small tensile strains while dentin regions near DEJ had bigger compressive strain. Moreover, concentrated tensile and compressive strains in outer dentin near the roots (in figure 9, marked with circle and triangles, respectively) which indicated the potential weakness in endodontically treated teeth after water loss. Another finding was that the unbound water loss induced deformations were also more prominent when compared to load induced deformations in the current experiment. However, since the sprayed speckles could decelerate, the totally exposed half-teeth sample would accelerate the full-field water evaporation. This finding should be validated again if in vivo experiments are performed.

6. Conclusions

Biomechanical behavior of dentin hard tissue in response to unbound water loss and mechanical loading was investigated in this paper. The accuracy verification of an adaptive high-magnification stereo-DIC system was considered in the unbound water loss experiments. The effects of speckles on the water evaporation were analyzed experimentally. Firstly, human dentin hard tissue is a heterogeneous, milky, white and porous composite that is filled with water. The speckles should attach to dentin suitably irrespective of dentin water evaporation. Thus, in our stereo-DIC experiments, black matte paint is used. Meanwhile, it is suggested that white paint should not totally cover the background. Otherwise, the human dentin surface will be an obstruction. Microscopic results showed that the sprayed speckles could adhere to the dentin surface well and would not totally obstruct the dentinal tubules, i.e., the sprayed speckles will decelerate full-field water evaporation. Secondly, unbound water loss of the dentin experiment needs long-term monitoring. Hence, heat balance of the adaptive high-magnification stereo-DIC system was considered. The cameras in the adaptive high-magnification stereo-DIC system were activated about 2 h prior to initiate preheating.

Based on the investigations of the effects of speckles on water evaporation and accuracy verification, the adaptive high-magnification stereo-DIC system was used to measure the biomechanical behavior of human dentin hard tissue in response to both partial dehydration and mechanical loading. It is anticipated that as in vivo dentin is wet and unbound water-loss of dentin is inevitable during endodontic treatment, testing of the mechanical properties (e.g. the elastic modulus, Poisson’s ratio) of dentin from a fully hydrated state to a non-hydrated state may help the biomechanical simulation of the tooth structure. Furthermore, this study demonstrated the performance of an adaptive high-magnification stereo-DIC system in the mechanical behavior measurement of a 3D fully/partially hydrated and heterogeneous dentin composite, which is a promising area of study.

The novelty of this paper is as follows: (1) the effects of speckles on water evaporation and measurement accuracy in the wet, curved and long-term measurement were analyzed; (2) the elastic modulus and Poisson’s ratio of both dentin and pulp in response to unbound water evaporation were measured simultaneously for the first time; (3) the full-field deformations of half teeth reveal that unbound water loss induced deformations were far more prominent when compared to load induced deformations.

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ORCID iDs

Xinxing Shao @ https://orcid.org/0000-0002-3112-7819
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