# Nanomechanical Properties of Endodontically Treated Teeth

*Robert A. Cheron, DMD, MS,* \* *Sally J. Marshall, PhD,*<sup> $\dagger$ </sup> *Harold E. Goodis, DDS,* \* *and Ove A. Peters, DMD, MS, PhD*<sup> $\star$ ‡</sup>

#### Abstract

Introduction: Although it is apparent that teeth become more susceptible to fracture after root canal treatment, the contributing factors for this are not completely established. The purpose of this study was to determine whether there are changes in nanomechanical properties of dentin in root canal-treated teeth compared with non-root canal-treated control teeth. Methods: Atomic force microscopy-based nanoindentation testing was performed on root canal-treated teeth and age- and type-matched control teeth. Radicular intertubular dentin was indented in 6 locations, and triplicate measurements were averaged. Paired t tests were used to compare root canal-treated teeth with control teeth. Results: The moduli of elasticity were 17.8  $\pm$ 2.9 GPa and 18.9  $\pm$  2.9 GPa for root canal–treated teeth and controls, respectively; the hardness values for the 2 groups were 0.84  $\pm$  0.25 GPa and 0.84  $\pm$  0.18 GPa, respectively. Neither the modulus of elasticity nor the hardness differed between groups (P > .05). Conclusions: It appears that root canal treatment does not result in nanomechanical changes to radicular intertubular dentin. (J Endod 2011;37:1562-1565)

#### **Key Words**

Dentin, elastic modulus, hardness, nanomechanical

This research was supported in part by a research grant from the American Association of Endodontists Foundation.

Address requests for reprints to Dr Ove A. Peters, University of The Pacific, Arthur A. Dugoni School of Dentistry, 2155 Webster Street, San Francisco CA 94115. E-mail address: opeters@pacific.edu

0099-2399/\$ - see front matter

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t is commonly believed that root canal-treated teeth are more prone to fracture than vital teeth (1). However, there has been no scientific demonstration that root canal-treated treated teeth have compromised mechanical properties and are more brittle or weaker than vital teeth; laboratory testing has revealed a similar rate of fracture between root canal-treated and nonendodontically treated teeth (2). Moreover, clinical studies indicate that root canal-treated teeth that did not receive cuspal coverage fail more often (3, 4). Specifically, over time, molars restored with direct composite and amalgam had a higher likelihood of failing (4, 5). These findings support the clinical recommendation for cuspal coverage after root canal treatment of molars but do not address the question whether loss of structure or changes in dentin properties are responsible for a higher rate of fracture after root canal treatment.

Teeth requiring root canal treatment are often structurally compromised as a result of caries, previous restorations, or trauma. Clinically, posts are placed into a root canal-treated treated tooth to help retain coronal buildups and full-coverage restorations. However, the preparation of a root canal-treated tooth to receive a post requires the additional removal of dentin and probably serves to further weaken the tooth, which may account for the increased occurrence of tooth fracture (6).

Dentin fracture patterns have been described as brittle (7); because of the loss of pulpal circulation, it was believed that dentin from root canal-treated teeth dried out (8) and became even more brittle. However, Sedgley and Messer (9) showed that there was only minimal moisture loss after root canal treatment. More recently, it became clear that dentin structure provides mechanisms to prevent crack propagation (10) and that fatigue is likely a possible mechanism of dentin fracture (11).

Atomic force microscopy (AFM) is a member of the scanning probe microscopy family of instruments (12, 13). In addition to providing high-quality images of a prepared dentin surface, AFM-based nanoindentation is useful for measuring mechanical properties, including hardness and elastic modulus of dentin. One of the major advantages of using the AFM for indentations as opposed to larger-scale indentation procedures is the ability to identify and test site-specific areas such as intertubular dentin.

A review of the literature reveals several studies (14–18) that show mechanical changes to dentin after exposure to various root canal irrigants, medicaments, and sealers; these studies all used segments of extracted human or animal teeth because of the impossibility of performing such an experiment *in vivo*. Methods of investigation ranged from 3-point bending tests to atomic force microscopy; the latter method appears to be superior because it accounts for the anisotropic nature of dentin behavior (19). AFM was not available for an earlier study assessing dentin hardness after root canal treatment (20); this study found no difference in Vickers hardness. This is a more global measure for hardness and does not differentiate between peritubular and intertubular dentin for example.

The different properties and behavior of dentin depending on location were noted previously (21-23). In addition, no data are available that assess the impact of root canal treatment on the nanomechnical properties of dentin. Therefore, this study aimed at determining the modulus of elasticity and nanohardness of teeth that have received root canal therapy and compared them with non-root canal-treated controls.

### **Materials and Methods**

#### **Specimen Collection**

Extracted human teeth that had previously received root canal treatment (RCT) along with control teeth were collected through the UCSF School of Dentistry's

From the \*Division of Endodontics and <sup>†</sup>Division of Biomaterials and Bioengineering, University of California San Francisco, San Francisco; and <sup>‡</sup>Department of Endodontics, University of The Pacific, Arthur A. Dugoni School of Dentistry, San Francisco, California.

Department of Oral and Maxillofacial Surgery and Division of Periodontology, local oral surgeons, and community clinics. The collection of teeth did not change the course of any patient's treatment plan and was performed according to the school's Institutional Review Board guidelines. Teeth were then examined using a stereomicroscope at  $3.5 \times$  to ascertain the absence of any root fractures or craze lines.

Care was taken to match teeth in both groups according to donor age at the time of extraction and tooth type, so that a total of 10 premolars, 2 canines, and 4 molars were included, with 5 pairs from the mandible and 3 from the maxilla. Immediately after collection, specimens were sterilized using  $\gamma$ -irradiation (24) and stored in deionized water and thymol until used. A power analysis had been performed to determine the number of specimens needed. Based on the sample size calculation ( $\alpha = 0.05$ , power 80%, minimum detectable difference of 25%, standard deviation of 15%), 8 teeth were used per group.

#### **Sample Preparation**

When multirooted teeth were used, the largest root available was used for sample preparation. This was either the distal root of a mandibular molar or the palatal root of a maxillary molar. Roots were sectioned at 3 and 7 mm from the apex, and the coronal and apical segments were removed. The remaining 4-mm-long segment was sectioned coronoapically through the center of the main root canal. The remaining buccal segment was then sectioned parallel to the first cut approximately one half the distance measured from the buccal surface to the root canal. The segment containing the root canal was used for data collection, and the other dentin samples were discarded.

Gentle gross polishing (through 1,200 grit) was followed by polishing using 10-, 5-, 1-, and 0.25- $\mu$ m diameter diamond polishing paste (Metadi; Buehler, Lake Bluff, IL). Specimens were then rigidly mounted on metal discs with polymethylmethacrylate for nanoindentation. Specimens were prepared in bulk at the start of the experiments and kept in Hanks' Balanced Salt Solution with phenol red indicator until used. The purpose of the phenol red indicator was to monitor the pH of the solution.

#### **Testing of Mechanical Properties**

The containers were monitored for pH changes on a weekly basis, and the storage solution was changed when pH changes from 7.4 to 7.2 and below were evident. Samples were removed from the storage solution and repolished with 1- and  $0.5-\mu m$  diamond paste to remove the surface layer that may have been altered by storage. The indentation procedures used in this study were described in detail previously (25). In brief, a Digital Instruments Nanoscope III AFM (Veeco, Santa Barbara, CA) and Triboscope head (Hysitron, Minneapolis, MN) were used to determine mechanical properties. A Berkovich diamond tip (triangular-based pyramid) was used for the indentations, which were performed in a liquid cell filled with deionized water at an ambient temperature in order to prevent alteration of mechanical properties because of desiccation (25). The maximum indenting load was 600 to 800 µN held over 3 seconds with loading and unloading rates of 200  $\mu$ N/s. Earlier studies had shown the collapse of dentin collagen upon dehydration (26, 27) and concluded that dehydration during measurements may induce measurement errors and thus should be avoided.

A standard of fused silica was used to calibrate the indenter before each day's measurements. A suitable 20  $\mu$ m  $\times$  20  $\mu$ m area of intertubular dentin was identified using AFM according to the following criteria (Fig. 14): intertubular dentin was evaluated for a level surface (no major changes in topography that may interfere with sampling) and the absence of scratches, indicating an appropriate surface polish.



**Figure 1.** Nanoindentation of dentin from root canal-treated (RCT) and control teeth. After visualization in the AFM, (*A*) a suitable intertubular dentin area for 6 indentations spaced about 3  $\mu$ m apart was selected (length bars are 5  $\mu$ m). Box plot diagrams show data for (*B*) elastic modulus and (*C*) hardness of intertubular dentin experimental and control teeth (n = 8 each). Every data point is averaged over 18 individual indentations.

Once an appropriate area of dentin was identified, 6 indentations were performed spaced about 3  $\mu$ m apart (Fig. 1*A*). Each sample was tested in 3 different locations of intertubular dentin; the modulus of elasticity and hardness for each sample were calculated from forces and displacements registered during indentations according to Oliver and Pharr (28), and each data point represents the average from 18 individual indentations per sample (Table 1).

#### **Data Collection and Analysis**

Data are presented as means and standard deviations; because the data were deemed to be normally distributed, paired *t* tests were used to determine any significant difference between the matched groups with  $\alpha = 0.05$  and power of 80%. To correlate mechanical properties with tooth donor age, Pearson correlation coefficients were also calculated.

#### Results

A total of 18 measurements for radicular intertubular dentin of each sample was collected and averaged (Table 1). The mean modulus of elasticity was 17.8  $\pm$  2.9 GPa and 18.8  $\pm$  2.9 GPa for root canal–treated teeth (donor age: 49  $\pm$  18 years) and control counterparts (donor age: 45  $\pm$  17 years) (Fig. 1*B*). Hardness was 0.84  $\pm$  0.25 GPa and 0.84  $\pm$  0.18 GPa for root canal–treated and control

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TABLE 1.	Elastic Mod	ulus and Hard	ness Average	d Over 18 Individ	ual
Indentatio	ons for Root	Canal-treated	(RCT) Teeth	and Age-matched	Controls

Group	Age (y)	Elastic modulus (GPa)	Hardness (GPa)
RCT	43	$13.7 \pm 4.4$	0.63 ± 0.24
	43	$19.4\pm2.0$	$\textbf{0.97} \pm \textbf{0.13}$
	43	$\textbf{18.9} \pm \textbf{2.0}$	$\textbf{0.98} \pm \textbf{0.11}$
	43	$\textbf{20.4} \pm \textbf{2.0}$	$\textbf{1.02} \pm \textbf{0.11}$
	43	$\textbf{20.8} \pm \textbf{3.6}$	$\textbf{1.15} \pm \textbf{0.20}$
	73	$\textbf{18.7} \pm \textbf{2.2}$	$\textbf{0.92} \pm \textbf{0.19}$
	25	$13.1\pm2.1$	$\textbf{0.47} \pm \textbf{0.07}$
	73	$\textbf{17.5} \pm \textbf{2.0}$	$\textbf{0.58} \pm \textbf{0.08}$
Mean $\pm$ SD	$49\pm16$	$\textbf{17.8} \pm \textbf{2.9}$	$\textbf{0.84} \pm \textbf{0.25}$
Control	41	$\textbf{21.2} \pm \textbf{2.8}$	$\textbf{1.00} \pm \textbf{0.13}$
	39	$\textbf{17.4} \pm \textbf{1.9}$	$\textbf{0.85} \pm \textbf{0.10}$
	42	$\textbf{20.3} \pm \textbf{4.7}$	$\textbf{0.88} \pm \textbf{0.26}$
	40	$\textbf{21.2} \pm \textbf{3.9}$	$\textbf{0.95} \pm \textbf{0.17}$
	39	$16.7\pm3.1$	$\textbf{0.61} \pm \textbf{0.21}$
	73	$\textbf{16.8} \pm \textbf{2.6}$	$\textbf{0.68} \pm \textbf{0.10}$
	19	$\textbf{22.6} \pm \textbf{5.5}$	$\textbf{1.10} \pm \textbf{0.22}$
	64	$14.2\pm2.0$	$\textbf{0.65} \pm \textbf{0.10}$
$\text{Mean}\pm\text{SD}$	$\textbf{45} \pm \textbf{16}$	$\textbf{18.9} \pm \textbf{2.9}$	$\textbf{0.84} \pm \textbf{0.18}$

Overall means and standard deviations (SD) are indicated. There were no significant differences comparing mean elastic modulus and hardness between RCT and control teeth.

specimens, respectively (Fig. 1*C*). Paired *t* tests indicated that there were no significant differences between root canal–treated teeth and control teeth for either elastic modulus (P = .593) or hardness (P = .997).

For control teeth, there were weak but significant negative correlations of donor age at the time of extraction with both modulus of elasticity and hardness ( $r^2 = 0.45$ , P < .05 and  $r^2 = 0.43$ , P < .05). No such correlations were found for dentin from root canal-treated teeth. However, when data points were plotted according to storage time, no systematic effect on mechanical properties was detected.

#### Discussion

This study sought to compare mechanical properties of age- and tooth type-matched radicular intertubular dentin with or without a history of root canal treatment. From this limited cohort of closely age-matched teeth, no difference in nanoscale elastic modulus and hardness was detected. Sample size is, although small, in keeping with other recent studies on mechanical properties of dentin using the AFM (21, 23). However, future studies may use larger cohorts with teeth subjected to identified irrigation solutions and post-root canal observation times.

As much as possible, teeth were prepared and tested in a standardized manner to minimize the influence of storage conditions or alteration in the testing setup. In order to minimize any effect of surface changes of the teeth because of prolonged storage of the thin sections after preparation (26), sample and control teeth were repolished and tested in an alternating pattern. Moreover, the age of the patient was known and root canal-treated and non-root canal-treated control teeth were extracted from similar age groups. However, another possible confounding variable is the time after root canal treatment, which was not known for most of the teeth included in the present study.

AFM (21–23, 25, 29) is increasingly used to detect differences in defined small areas of potentially anisotropic composite materials, such as dentin. We report an elastic modulus for intertubular dentin that is similar to that reported both in earlier (22) (17.7-21.1 GPa) and current (29) (17 GPa) studies. Lower values had been determined

by Poolthong et al (23) (14.9 GPa), but that study focused on a differently shaped indenter and used a higher maximum indentation force.

The technique of nanoindentation allows the measurement of local moduli of elasticity using a very fine tip. The Berkovich tip used in this study is pyramidal in shape, with a length and width of roughly 6  $\mu$ m. There is concern that repeated sampling results in degradation of the fragile tip and altered readings. However, plotting the data for modulus of elasticity and hardness in chronological order revealed no trend in the data because of the deterioration of the tip. To overcome desiccation occurring when dry specimens are indented, the triboscope used in our laboratory allowed experimentation with fully hydrated specimens (25). In addition, the tip was used to indent a standard of known hardness and the modulus of elasticity before each day's measurements; all such readings were normal.

Prior studies have examined the previous effects of irrigants and root canal therapy on the hardness of teeth (9, 30, 31). The particular irrigants used during the endodontic treatments of the teeth included in this study are not known; however, standard deviations within each sample and also among the samples were low. Furthermore, the patient age, or more specifically the tooth, may be correlated to higher fracture susceptibility (19, 32, 33). In the present study, we identified a weak trend toward lower moduli of elasticity in teeth from older patients and without root canal treatment, which could correlate with the observed reduced fracture toughness of dentin with age (34). A possible avenue for future research may use a larger sample size with a broader age distribution of patients to address the potential age dependency of dentin nanohardness in detail.

It appears that it is still not finally resolved if and potentially how reduced fracture toughness is associated with changes of dentin moisture content after root canal treatment (35-37). Other factors for fracture susceptibility of root canal-treated teeth are the loss of structure from the access cavity and canal preparations, the use of aggressive irrigation solutions and inappropriate restorations (1).

In conclusion, from the data collected in this study, there appears to be no difference in the modulus of elasticity or the hardness of radicular intertubular dentin when comparing root canal-treated and control teeth. Although caution should be exercised to extrapolate this initial study, the findings may suggest that changes in mechanical properties of intertubular dentin are unlikely to be causative for fractures after endodontic treatment.

#### Acknowledgments

The authors thank Grace Nonomura and Dr Kuniko Saeki for their expert technical assistance.

The authors deny any conflicts of interest related to this study.

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