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# Characterization of the elastic and viscoelastic properties of dentin by a nanoindentation creep test

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ABSTRACT

Dentin is the main supporting structure of teeth, but its mechanical properties may be adversely affected by pathological demineralization. The purposes of this study were to develop a quantitative approach to characterize the viscoelastic properties of dentin after de- and re-mineralization, and to examine the elastic properties using a nanoindentation creep test. Dentin specimens were prepared to receive both micro- and nano-indentation tests at wet and dry states. These tests were repeatedly performed after demineralization (1% citric acid for 3 days) and remineralization (artificial saliva immersion for 28 days). The nanoindentation test was executed in a creep mode, and the resulting displacement–time responses were disintegrated into primary (transient) and secondary (viscous) creep. The structural changes and mineral densities of dentin were also examined under SEM and microCT, respectively. The results showed that demineralization removed superficial minerals of dentin to the depth of 400 μm, and affected its micro- and nano-hardness, especially in the hydrate state. Remineralization only repaired the minerals at the surface layer, and partially recovered the nanohardness. Both the primary the secondary creep increased in the demineralized dentin, while the hydration further enhanced creep deformation of untreated and remineralized dentin. Remineralization reduced the primary creep of dentin, but did not effectively increase the viscosity. In conclusion, water plasticization increases the transient and viscous creep strains of demineralized dentin and reduces load sustainability. The nanoindentation creep test is capable of analyzing the elastic and viscoelastic properties of dentin, and reveals crucial information about creep responses.



## 1. Introduction

Natural teeth are function-oriented bilayer composites, with dentin constituting the main part to support the overlying enamel. While the outer enamel exhibits higher stiffness, the inner dentin absorbs substantial impacts and stresses (Goel et al., 1991; Jung et al., 1999). Investigations into dentinal mechanical properties are significant in realizing its physiological functions and pathological changes, and in assessing repair materials. Since the samples used in such works are generally small, microindentation tests are widely used to examine the elastic properties of dentin (Xu et al., 1998). However, these tests measured dentin as a homogeneous solid, thus limiting the characterization of an ultrastructure. With an equipped atomic force microscopy, nanoindentation tests may examine inhomogeneous and anisotropic dentin (Han et al., 2012; Kinney et al., 1999), and provide more details about its structure-mechanical functions.

Erosion is a common problem that leads to local or extensive demineralization of dentin. Demineralization primarily removes the extrafibrillar minerals of dentin, especially at the superficial layer (Breschi et al., 2002). However, traditional approaches use dry dentin, and the mineral-depleted collagen matrixes on such specimens may collapse and lead to overestimated stiffness (Bertassoni et al., 2010). Examination of hydrated dentin is thus important with regard to the altered mineral content. Additionally, experimental models of erosive dentin usually include the remineralization to clarify the reversed structural changes and mechanical properties.

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Recent revisions to the classical remineralization by saliva or artificial analogs showed epitaxial deposition of minerals instead of intrafibrillar crystallization (Kinney et al., 2003; Tay and Pashley, 2008). Since remineralization by natural saliva is a basic physiological function, there remains a need for particular investigations.

As dentin absorbs stress, its viscoelastic properties are more responsible for its ability to dissipate energy compared to its elastic properties. Dentin has linear-viscoelastic behavior (Balooch et al., 2004; Jantarat et al., 2002), with hydration enhancing its stress–relaxation behavior (Trengrove et al., 1995). Demineralized dentin has high creep rates (Pashley et al., 2003), which result into viscous deformations under low loads in regular functioning (Singhal et al., 2013).

Nanoindentation creep tests have been used to explore the viscoelasticity of biological tissues using a prolonged load-holding time (Shepherd et al., 2011). The resulting measurements can be converted into viscoelastic parameters by combining existing mechanical models, and applied in the extending analysis of plastic deformation and damage. Among various rheological models, Burgers viscoelastic model is more accurate in characterizing the mechanical behaviors of bone tissue, irrespective of loading rate and type (Wu et al., 2011). By using this model, the data from laboratory creep tests can be extrapolated to predict long-term behavior. However, the combination of a nanoindentation creep test and mechanical modeling has not yet been applied in measuring dentinal viscoelasticity. Accordingly, this study aimed to investigate the elastic and viscoelastic properties of de- and re-mineralized dentin, and to characterize the viscoelasticity of dentine by a nanoindentation creep test and Burgers model.

## 2. Materials and methods

The study protocol was approved by the Institutional Review Board, National Cheng Kung University Hospital. Twenty sound extracted human molars were collected with informed consent, and then sectioned to generate eighty dentin slabs ($4 \times 4 \times 2$ mm$^3$). Slabs from the same tooth were divided into two groups that underwent either the micro- or nano-indentation tests. The buccal sections of tooth crowns were preserved for the measurement of mineral density. Fig. 1 shows the experimental scheme of this work.

### 2.1. Microindentation test

The dentin specimens were individually embedded in epoxy resin with their top surfaces exposed, then serially polished with 1500 grit sandpapers, and finished with 1 μm diamond suspension. The mechanical tests were performed at both hydrated and dry states. The slabs were first kept in deionized water for 24 h before the mechanical tests.

The microindentation tests were conducted with a microhardness tester (HMV II, Shimadzu, Tokyo, Japan) at a load of 25 g and dwell time of 5 s using a Vickers indenter. At least five readings at different locations were taken for each test.

The Vickers microhardness ($Hv$) of dentin was thus calculated as follows:

$$Hv = \frac{0.1891\,F}{d^2} \tag{1}$$

where $F$ is the set indentation force in N, and $d$ is the length of the pyramid diagonal in mm. For comparisons of micro- and nanoindentation tests, $Hv$ was converted into microhardness ($\mu H$) in MPa by multiplying it by 9.807.

### 2.2. Nanoindentation test for elastic constants

The nanoindentation tests were conducted using a computer-controlled nanoindenter (TI 700 UBI, Hysitron, MN, USA) equipped with a Triboscan system (Hysitron, Minneapolis, MN, USA). A Vickers diamond indenter with a radius of 50 nm was used, so the $\mu H$ and $nH$ values could be compared. A trapezoidal force profile was applied, with the force increasing at a speed of 100 μN/s to the peak force of 1000 μN in 10 s, held for 10 s, and finally unloaded in 10 s (Fig. 2A). The indentation test in the hydrated state was performed in a liquid cell filled with deionized water, while the test in the dry state was carried out in air. During indentation, the applied load $P$ and displacement $h$ were continuously monitored and recorded.

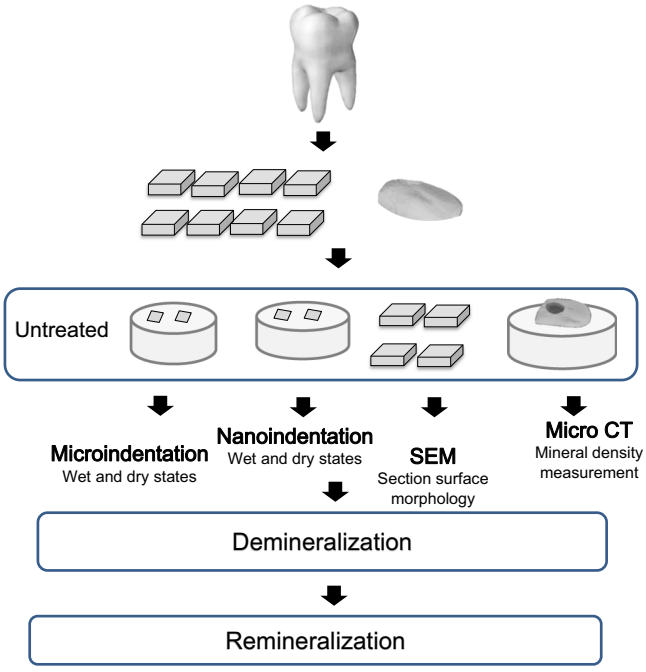


**Fig. 1.** Experimental design for the mechanical, morphologic, and mineral density examinations of dentin.

The measurement of the contact stiffness depends on the derivative of the unloading curve at the initial point (Oliver and Pharr, 1992) as

$$S = \frac{d\,P}{d\,h} \tag{2}$$

Since this investigation used a creep loading mode instead of the conventional loading–unloading procedure, the displacement affected by creep was corrected to derive the corrected contact stiffness $S_c$. The relationship between stiffness and elastic modulus (Doerner and Nix, 1986) may be defined as

$$S_c^{-1} = S^{-1} - \frac{(d\,h/d\,t)}{(d\,P/d\,t)} = \frac{2n}{\sqrt{\pi}}\sqrt{A}\,E_r \tag{3}$$

where $n = 1.012$ is a correction factor for the Vickers indenter, $A$ is the projection of the contact area, and $E_r$ is the reduced elastic modulus. The elastic modulus $E$ of dentin can be derived as

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{\left(1-\nu_i^2\right)}{E_i} \tag{4}$$

$\nu$ and $\nu_i$ are the Poisson ratios of the specimen and the indenter, respectively. $E_i$ is the elastic moduli of the indenter. Since the $E_i$ (1140 GPa) is significantly greater than $E$, and $\nu$ is quite small ($-0.025$ to 0.26) (Renson and Braden, 1975), $E$ can be approximated by $E_r$.

### 2.3. Modeling the viscoelasticity of dentin

The strain–time curves were extracted to fit the Burgers model and thus model dentinal viscoelasticity. The Burgers model is a four-element model, in which a Maxwell unit and Kelvin unit are connected in series (Fig. 2B). The constitutive equation for the Burgers model is defined by setting the strain response $\varepsilon$ as the sum of the strains of three elements (Yang et al., 2006).

$$\varepsilon = \varepsilon_{Ms} + \varepsilon_K + \varepsilon_{Md} \tag{5}$$

where $\varepsilon_{Ms}$, $\varepsilon_K$, and $\varepsilon_{Md}$ indicate the stains of the Maxwell spring, Kelvin, and Maxwell damper unit, respectively. The creep behavior along the loading time can thus be obtained as follows:

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