Mechanical properties of human dentin

Part I – Measurement of elastic modulus and damping by mechanical spectroscopy

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Abstract— **Human dentin exhibits a mechanical behavior like a functionally graded material with properties dependent on many physical and biological factors. This work describes and critically analyzes the methods to measure elastic moduli and damping and the corresponding results reported in literature. Furthermore, Mechanical Spectroscopy (MS) experiments have been carried out to determine with high precision the values of Young's modulus and Q^{-1}.**

Keywords— ***dentin, measurement, Young's modulus, damping, mechanical properties, mechanical tests***

I. INTRODUCTION

Dentin is a hard, elastic and avascular tissue forming the tooth bulk and supporting the enamel. It contains principally hydroxylapatite (HAp) and organic material, in addition to water: it is a particular form of calcified tissue produced by odontoblasts which are arranged in a continuous layer along the pulp cavity. Dentin has a striated appearance due to the presence of dentinal tubules radiating from the pulp cavity to the outer surface and it is organized in microscopic channels (tubules). Their distribution, density and orientation vary with the position [1].

Dentin exhibits elastic, anelastic and plastic behavior and the knowledge of its properties is essential in clinical dentistry for understanding the effects of various restorative dental procedures and for predicting the effects of microstructure alterations due to caries, sclerosis and aging on tooth strength. This work reviews and critically discusses data on elastic and anelastic properties reported in literature and presents results obtained by Mechanical Spectroscopy (MS).

II. LITERATURE DATA

A. E and G elastic moduli

TABLE I. lists data of *E* and *G* moduli available in literature; experimental techniques and other details are also reported. Tests are commonly performed by static (compression, flexure) and dynamic (DMA, ultrasound) techniques in order to obtain a mean value of elastic properties. On the contrary, indentation provides local values of Young's modulus thus can be used for mapping the elastic properties of human teeth. Mean values of elastic constants are often obtained by compression tests carried out both on cylindrical (Φ = 2.5-3.5 mm, h = 2.5-10 mm) and brick (w =1.5-3 mm, d = 1-2.5 mm, h = 2.5-10 mm) samples cut from a single tooth.

TABLE I. E AND G ELASTIC MODULUS

Ref.	E and G (GPa)	Notes	Technique
[25]	E = 29.8	Peritubular dentin	Nano-indentation with Berkovitch punch
	E = 17.7 ÷ 21.1	Intertubular dentin	
[26]	E = 28.6 – 34.2	Peritubular dentin	Nano-DMA
	E = 18.1 - 21.6	Intertubular dentin	
[16]	E = 19.5 ÷ 26.5	E map	Nano-indentation with Berkovitch punch
[23]	E = 6.5 - 38.1	E map	Knoop imprint dimension
[20]	E =19.65	Mean value	Nano-indentation on Atomic Force Microscope (AFM) system
[20]	E = 17 - 23	Mean value	Nano-indentation on Atomic Force Microscope (AFM) system
[17]	E = 19.89 ± 1.92	Map in primary dentin	Ultra-Micro-Indentation-System (UMIS) with Berkovitch punch
[18]	E = 11.59 ± 3.95	In primary dentin near pulp	Ultra-Micro-Indentation-System (UMIS) with Berkovitch punch
	E = 17.06 ± 3.09	In primary middle crown dentin	
	E = 16.33 ± 3.83	In primary DEJ	
[27]	E = 0.013 ÷ 23.1	In carious dentin	Ultra-Micro-Indentation-System (UMIS) with Berkovitch punch
[22]	E = 20	Mean value	Nano-indentation on Atomic Force Microscope (AFM) system
[19]	E = 10.1 ÷ 19.3	Map	Nano-indentation
[29]	E = 1.9 ÷ 2.3	Demineralized dentin (like soft tissue)	Nano-indentation on Atomic Force Microscope (AFM) system
[7]	E= 14.46±2.49	Mean value (different geometry of samples)	Three-point flexure test
[8]	E = 15.0 ± 0.5	Mean value	Three-point flexure test
[9]	E = 18.7 ± 3.5	Mean value for θ = 0°	Four-point flexure test
	E = 15.5 ± 2.8	Mean value for θ = 90°	
[10]	E = 12.8 ÷ 14.6	Mean value	Four-point flexure test
[1]	E = 10.4 ± 2.9	Mean value	Compression test
[3]	E = 13.3 ± 1.3	Mean value	Compression test
[4]	E = 16.1	Mean value	Compression test
[5]	E = 11.5	Mean value	Compression test
[6]	E = 13.26 ± 1.8	Mean value	Compression test
[35]	$E = 19 \pm 5\times10^{-6}$	Mean value	Mechanical Spectroscopy
[31]	G = 5.77 ÷ 11.6	Mean value	Torsion pendulum
[11]	E = 14.3 ÷ 15.8	Mean value	Dynamic Mechanical

Ref.	E and G (GPa)	Notes	Technique
			Analysis (DMA)
[12]	E = 24 ± 1	Mean value	Piezoelectric ultrasound system
[15]	E = 26.5	Mean value	Resonant Ultrasound Spectroscopy (RUS)
	G = 10.3	Mean value	
[14]	G = 8	Mean value	Resonant Ultrasound Spectroscopy (RUS)
[13]	E = 28.3	Mean value	Resonant Ultrasound Spectroscopy (RUS)
	G = 8.6 ÷ 11.1	Mean value	

E values from literature [1]-[7] obtained by compression tests range from 10.4 to 16.6 GPa.

The influence of sample geometry on experimental results is considered in [7]. As shown in Fig. 1 dentin exhibits various types of response from brittle to highly deformable under compression depending on the geometry of the sample (d/h is the ratio of brick sample). Elastic and plastic deformations and ultimate compressive strength increase with d/h ratio whereas Young's modulus decreases. Consequently, samples with the maximal d/h ratio exhibit the highest elasticity and plasticity, but the smallest Young's modulus.

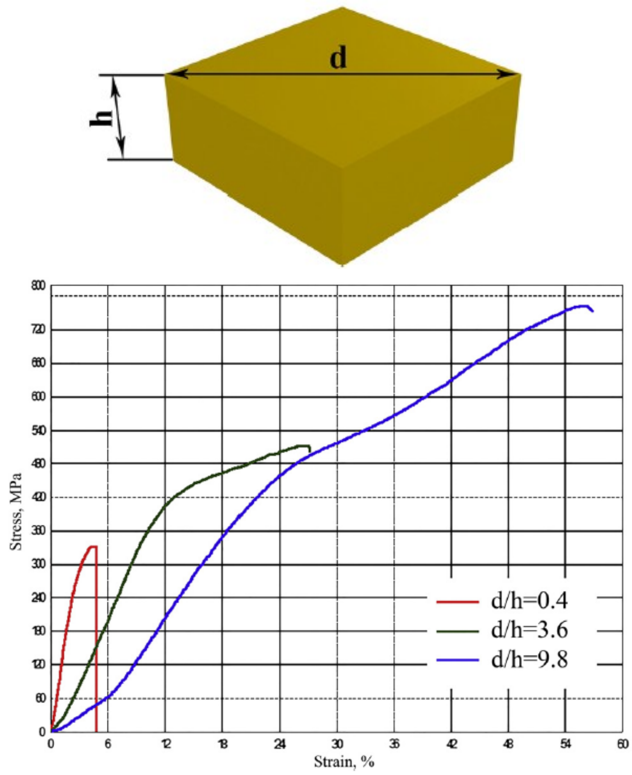

Fig. 1. Compression curves of dentin sample varying specimen geometry [7]

The effect of shape is not relevant in three point bending tests. In fact, three point and four-point flexural tests are commonly used and provide Young's modulus data similar to those from compression tests, as in [8]-[10]. From the analysis of results obtained in static tests (compression and flexural) relevant data scattering of about ± 20% is observed.

In the case of dynamic methods, tests are carried out by means of various techniques operating in different conditions, in particular frequency may vary in an extended range from 0.1 Hz to several GHz. Three main types of tests can be identified: a) sub-resonance tests, b) resonance tests and c) wave propagation tests.

Experiments employing the Dynamic Mechanical Analyser (DMA) are described in [11]. The instrument operates in sub-resonance conditions with forced vibrations from 0.1 Hz to 10 Hz. The specimen is clamped as a single cantilever beam in the mounting frame of the machine that digitally generates a sinusoidal flexural stress applied to the specimen via a small vibrator. The specimen movement is measured by a linearized eddy current displacement transducer and its signal is processed to get the complex dynamic modulus. The value *E* = 15 GPa has been obtained.

Ultrasound techniques employ the piezoelectric excitation of cylindrical samples at high frequency [12] and Young's modulus is determined through the measurement of the resonance frequency. The values are in general a little higher than those obtained from other techniques.

Among ultrasound techniques it is noteworthy to mention RUS (Resonant Ultrasound Spectroscopy). Hooke's law and Newton's second law permit to predict the resonant modes of mechanical vibrations of a specimen of known shape. From the resonant modes, all of the elastic constants can be uniquely determined from a single measurement if the density of the specimen is known. Values of *E* ranging from 24 to 28.3 GPa and of *G* from 8 to 11.1 GPa are reported in [13]-[15]

Comparing experimental results from different techniques, it can be observed that data scattering in ultrasound measurements (± 8 % and ± 16 % for E and G respectively) is lower than that from static tests.

To obtain a local characterization of elastic constant, instrumented micro- and nano-indentation is largely used to carry out local measurements on teeth. Maps of elastic modulus are reported in [16]-[18] employing nano- and micro-punches of different type, such as Vickers, Knoop and Berkovitch. Mean values of *E* are given in [20]-[22].

An indirect measurement of *E* has been made by Knoop micro-hardness tests [23]. This method is based on the concept that the length decrease of the imprint diagonals due to elastic recovery can be related to the hardness-modulus ratio. Therefore, *E* can be determined from the relationship:

$$E = \alpha_1 HK/(b/a - b'/a') \qquad (1)$$

where α_1 = 0.45 is a constant determined experimentally, HK is the Knoop hardness, *b*/*a* and *b'*/*a'* are the ratios between imprint diagonals at full load and after elastic recovery, respectively.

Analysis of literature data shows that *E* value strongly depends on the position. The most relevant variation is along a radial direction from the enamel to the pulp as reported in [20] and [21]: the highest value is near DEJ and the lowest one near the pulp cavity. This spatial gradient of elastic modulus is due to the propagation of cracks from enamel into dentin [22]: in fact, enamel is a typical hard and brittle material whereas dentin is a tougher biological composite.

Data from nano-indentation tests reported in literature give a mean value E = 21 GPa (± 4,7%). For the local characterization of elastic properties nano-indentation shows a serious drawback, i.e. the imprint size is comparable to the tubule section. In addition, surface roughness strongly affects the results. Since the technique provides data affected by a large scattering, a very great number of tests is necessary to get reliable values. Therefore, micro-indentation seems to be more suitable for mapping local mechanical constants [23]. Conversely, nano-indentation can be usefully employed to determine peri- and inter-tubular properties.

Dentin has a highly ordered microstructure that can be modeled as a continuous fiber-reinforced composite, with the intertubular dentine forming the matrix and the tubule lumens with their associated cuffs of peritubular dentine shaping the cylindrical fiber reinforcement as described in [24] and [25]. The highest value is related to peritubular region (~ 29 GPa) with a variation related to the position, while mean Young's modulus in intertubular dentin is about 19 GPa. Results are confirmed also in [26] where elastic modulus is measured by nano-DMA. With this technique E is obtained by superposing a sinusoidal load on the static contact load during a dynamic nanoindentation.

Tubules orientation is another structural factor influencing the elastic modulus. If one considers a beam-shaped sample, tubules can be perpendicular (θ = 0°) or parallel (θ = 90°) to the sample length. It has been demonstrated that elastic modulus is higher for θ = 0° [27].

This anisotropy of dentin is described also in [28] where the relationship between deformation behavior under shear test and the orientation of dentin tubules has been analyzed. In particular, experimental measurements are carried out considering different scenarios, as shown in Fig. 2: 1- dentin tubules lie both parallel to the plane of shift and perpendicular to the direction of loading (ZY plane, x load), 2- tubules lie both perpendicular to the plane of shift and perpendicular to the direction of loading (XY plane, y load) and 3- the dentin tubules are oriented both parallel to the plane of shift and parallel to the direction of loading (ZX plane, z load).

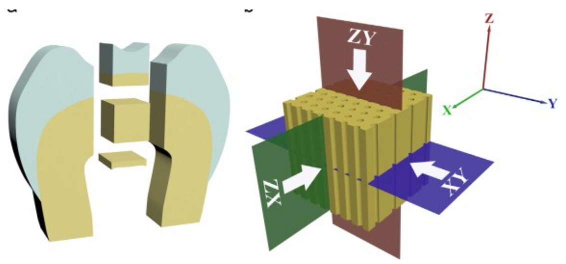

Fig. 2. Planes and direcion of shift [28]

Deformation behaviour and shear modulus of the dentin samples, where dentin tubules are parallel to the plane of shift and parallel or perpendicular to the direction of loading, is the same (scenario 1 and 3). On the other hand, for scenario 2 where dentin tubules are perpendicular to the plane of shift and perpendicular to the direction of loading, shear modulus is lower than other groups of samples and the fracture is easy in the plane normal to the dentin channels.

The mechanical properties of dentin are also related to its mineral content as described in [19], [27]-[30]; for instance, it is well known that E decreases in carious dentin and after a complete demineralization in EDTA dentin becomes similar to a soft tissue and E value is about 2 GPa [29]. In addiction, mineral content is relate to age. The influence of mineral content on elastic characteristics is important because structural variations of normal dentin prejudice the healing process in restorative and preventive dental treatments. Differences are revealed also by considering primary dentin as in [17], [18].

From indentation results G can be calculated through the relationship:

$$G = E / 2(1+\nu) \qquad (2)$$

Taken ν = 0.29 [13], indirect values of G can be obtained from E data listed in the previous table; the average value of 7 GPa is in good agreement with values determined by direct measurements [13]-[15], [31].

B. Damping

If a solid exhibits an hysteretic loop along a load-unload cycle, stress is out of phase with strain and energy loss occurs. The energy loss, which is the physical origin of damping, depends on the off-phase angle δ. The loss factor, $\tan\delta$, describes the amount of energy stored/returned by a sample during a single load-unload cycle and provides a measure of damping. Loss factor, also referred as Q^{-1}, is often so small that it is not possible to get it by a direct measurement of the angle δ, in this case Q^{-1} is determined from the logarithmic decay d of flexural vibrations:

$$Q^{-1} = \tan\delta = d/\pi \qquad (3)$$

Damping measurement tests can be performed through different experimental techniques and devices operating in an extended range of frequencies from about 0.1 Hz to several GHz. In general four types of tests can be identified: 1- quasi-static tests; 2- sub-resonance tests; 3- resonance tests; 4- wave propagation tests.

Quasi-static tests are carried out by using conventional tensile machines and measuring hysteretic loops under constant strain rate ($\pm\, d\varepsilon/dt$).

In sub-resonance tests the samples are forced to vibrate at frequencies very low with respect that of resonance and the loss angle δ is detected. The technique is largely employed for testing teeth and several commercial instruments, dynamic mechanical analyser (DMA), are available.

The tests performed in resonance conditions are commonly made on wires or sheets but can be carried out also on samples of different and complex shape by means of a vibrating hammer. The last method has been employed for *in vivo* tests on human teeth.

In the tests based on wave propagation short pulses at high frequency (up to several GHz) are sent through the sample to measure wave speed and attenuation. From the ultrasound speed the elastic modulus is determined, from the attenuation coefficient Ξ the Q^{-1} value:

$$Q^{-1}= \Xi\lambda/\pi \tag{4}$$

being λ the ultrasound wavelength.
Table II reports literature data obtained by different methods and in different conditions.
At room temperature, sub-resonance [32],[34] and resonance [35] tests on dentin give values of damping (Q^{-1} = 0.01) similar to those found for human bone ranging from 0.02 to 0.04 [33].
Damping is affected by many factors, in particular it increases if dentin is damaged and structural defects are present [33]. As shown in [32], also the reduction of mineral content by means of immersion in EDTA solution induces an increase of damping.
Huang et al. [36] carried out measurements *in vivo*. The experiments were performed on 15 volunteers and their teeth were previously examined by X-Ray imaging. Incisor teeth were forced into vibration by the application of an impulse force hammer on the surface of tooth in the lingual-labial direction and an acoustic microphone was used as transducer. The vibration signal was then transferred to a frequency spectrum analyzer for resonance frequency and damping ratio display. The very large Q^{-1} value (~ 0.14) was attributed to the damping effects of periodontal ligament and natural tissue around teeth. However, experiments, repeated *in vitro* [37] by the same investigators, provided analogous results indicating that the method of vibrating hammer is not very sensitive and can provide only a rough estimation of damping.
Finally, it is noteworthy the completely different approach for the measure of loss factor that has been proposed by Sakamoto et al. [43]. These investigators used dynamic indentation to get the values of damping on a local scale.
The same approach is presented in [7] and [39]. Results from the nano-DMA show that there is a significant distinction in damping between intertubular and peritubular dentin, as reported in Table II.

TABLE II. DAMPING

Ref.	Damping (tan δ)	Notes	Technique
[32]	0.01 – 0.03	For T = 0 ÷ 300°C	DMA
	0.03 – 0.08	For T = -50 ÷ 80°C	
[33]	0.05 ± 0.01	Influence of structural defects	DMA
[34]	0.04	Mean value for T = 37-200°C	DMA
[35]	0.01	At isothermal test	Vibrating Reed Analyzer (VRA)
[36]	0.146 ± 0.037	Damping *in vivo*	Vibrating hammer *in vivo*
[37]	0.144 ± 0.022	Damping *in vitro*	Vibrating hammer *in vitro*
[38]	0.08 – 0.1	Damping on local scale	Dynamic nano-indentation
[7]	0.015 – 0.065	Peritubular dentin	nano-DMA
	0.012 – 0.053	Intertubular dentin	

Moreover, experimental measurements of tanδ show important differences in the dampening behavior between the young and old dentin as function of collagen degradation and mineralization of intertubular spaces with aging [39]. Nevertheless this technique presents a high scattering of experimental data, in particular results strongly depend on frequency of dynamic load and on the imprint size comparable with tubule dimension.

III. EXPERIMENTAL RESULTS

To overcome some problems typical of other techniques measurements of dynamic modulus and damping have been carried out by Mechanical Spectroscopy (MS).
Human molars were extracted from individuals (males 55-70 years old) as part of their dental treatment. After disinfection by immersion in a solution of sodium hypochlorite in water for about 12 hours, they were longitudinally sectioned in order to obtain 0.8 mm-thick slices. From these sections bar-shaped samples (length L = 13÷16 mm) have been cut for MS measurements. The specimen size involved a certain degree of non homogeneity of dentin characteristics since they included root dentin and crown dentin. A number of 15 teeth taken from different patients (one tooth per patient) have been used in the experiments and a single specimen was obtained from each tooth. Dentin density is different from point to point decreasing from the outer part to the inner one thus mechanical properties are not homogeneous. Therefore, elastic modulus E and damping factor Q^{-1} from present experiments represent average values.
Before testing the specimens have been investigated by scanning electron microscopy and light optical microscopy to reject those with fractures or damages.
The Vibrating Reed Analyzer VRA 1604 apparatus employed in the experiments (Fig. 3) was described in detail in [40].

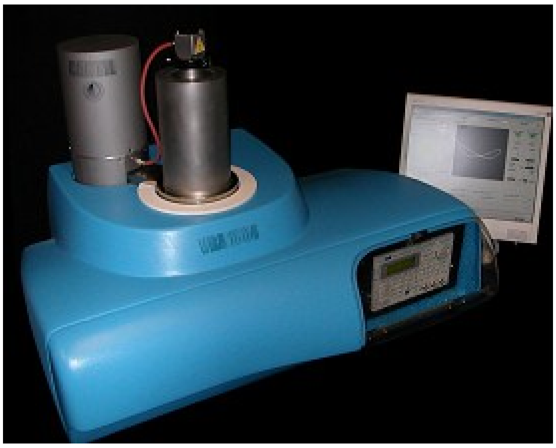

Fig. 3. VRA apparatus employed in the experiments.

The samples, mounted in free-clamped mode, have been tested using the method of frequency modulation.
In Fig. 4, a typical bar-shaped sample mounted in the sample holder is shown. One side of the sample is covered by a very thin gold layer (~ 2 μm) because flexural oscillations are induced by an electrode parallel to the sample.

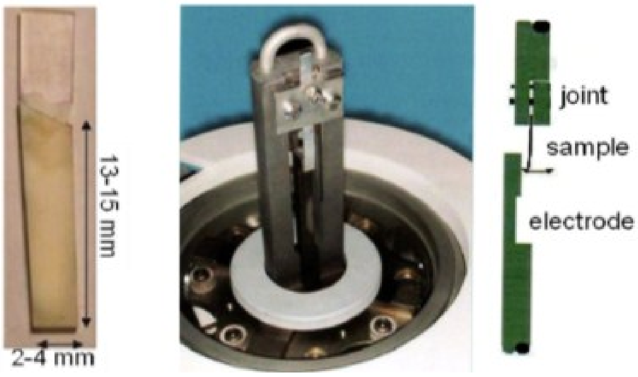

Fig. 4. Samples mounted on VRA

Q^{-1} values have been determined from the logarithmic decay *d* of flexural vibrations (Fig. 5):

$$d = (1/k)\ ln\ (A_n/A_{n+k}) \qquad (5)$$

being A_n and A_{n+k} the amplitudes of the *n*-th and *n+k*-th oscillation. Q^{-1} is calculated by:

$$Q^{-1} = d/\pi \qquad (6)$$

The mean Q^{-1} value determined in present experiments is 0.01 with data scattering of about 2 %.

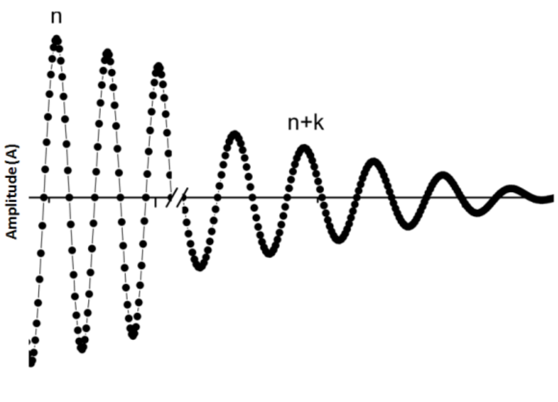

Fig. 5. Logaritmic decay *d* of flexural vibration

Dynamic modulus *E* was obtained from the resonance frequency *f* by:

$$E = f^2\ (48\pi^2 PL^4)/(m^4 h^4) \qquad (7)$$

where *m* is a constant (*m* = 1.875), *P* the material density, *L* and *h* the length and thickness of the sample. Strain amplitude was kept lower than 1 x 10^{-5}; test frequencies were in the range of kHz.
The value *E* = 19 GPa was determined in present experiments. Data scattering of measurements carried out on 15 samples was 1 %.

IV. CONCLUSIONS

The work describes the experimental techniques to measure elastic properties and damping behavior of human dentin. Literature data have been presented and discussed by considering the effects of several parameters, such as patient age, dentin mineralization and hydration, tubules orientation with respect the applied load etc..
Mechanical Spectroscopy experiments have been carried out on 15 different samples and the average values *E* = 19 GPa and $Q^{-1} = 0.01$ determined by means of this dynamic technique show a scattering of. 2 % for damping and 1 % for elastic modulus. Data scattering is much smaller than that of experimental methods.

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