Mechanical properties of human dentin

Part II – Measurement of local characteristics by FIMEC indentation test

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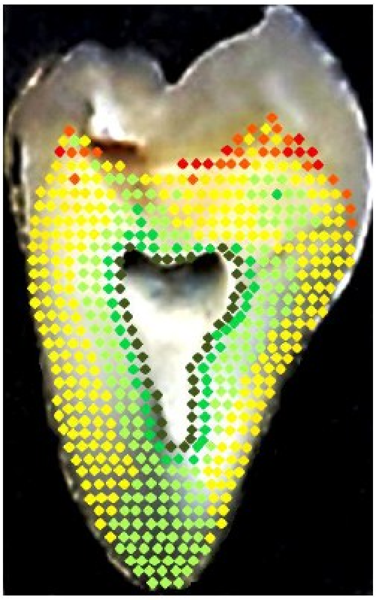
***Abstract*- The mechanical characteristics of dentin have been investigated on local scale by the instrumented indentation test FIMEC employing a cylindrical punch. The technique permitted to measure in different tooth positions the elastic modulus, yield stress, stress-relaxation and creep.**
The punch diameter (Φ = 0.5 mm) is much larger than the tubule size thus data are not so largely scattered as in micro- and nano-indentation tests but, at the same time, is small enough to guarantee a good resolution in mapping the mechanical properties. The results are in good agreement with literature data obtained by means of various experimental techniques.
Furthermore, a new algorithm has been developed for analysing the experimental indentation curves in view of the realization of a commercial FIMEC apparatus.

***Keywords*- Dentin, instrumented indentation, FIMEC, local mechanical characterization**

I. INTRODUCTION

Dentin is a calcified tissue of the body, and along with enamel, cementum and pulp is one of the four major components of human teeth. In a natural tooth, it is covered by enamel on the crown and cementum on the root and surrounds the entire pulp. One of the main characteristics of dentin in human teeth is the presence of dentinal tubules radiating from the pulp cavity to the outer surface with distribution, density and orientation depending on the position [1-3].



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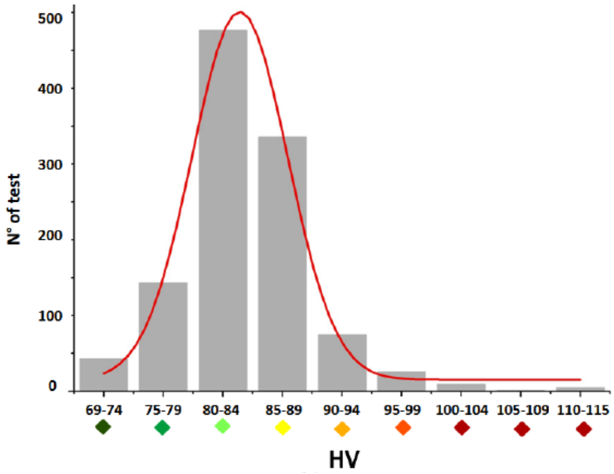

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Fig. 1. Map of dentin Vickers micro-hardness (a) and Gaussian data interpolation (b) [4].

Both the morphology of dentinal tissue and the anisotropy of its structure affect the mechanical properties on local scale, as clearly shown in the Vickers micro-hardness map of a tooth section obtained by Cappelloni [4] and reported in Fig. 1 (a-b). The result confirms the dependence of mechanical properties from position and in particular the hardness gradient from enamel to pulp along radial direction.
This aspect is fundamental in clinical dentistry because the knowledge of local dentine properties as a function of the position is very important for understanding the effects of the wide variety of restorative dental procedures from the design of preparations to the choice of bonding methods. To obtain this goal, in the last years nano-indentation has been widely applied to measure Young's modulus of mineralized tissues and other biomaterials [5-11]. Nevertheless, the technique provides data affected by a large scattering because the imprint size is comparable to that of tubule sections. In addition, surface roughness is an unavoidable drawback when one operates on a nano-scale.
The present work focused the attention to the development of a reliable methodology based on instrumented indentation for the local mechanical characterization of dentine in different

tooth positions. FIMEC (Flat-top cylinder Indenter for MEchanical Characterization) is an indentation technique, developed by one of the authors [12-14], which employs a cylindrical punch of sintered tungsten carbide WC ($\nu = 0.24$, $E = 668$ GPa). It permits to determine yield stress, Young's modulus, stress-relaxation and creep behaviour on local scale. In the past it has been successfully used for investigating different types of metals [14]. The experimental apparatus, described in detail in [13], has been suitably modified to operate with lower applied loads and tests have been performed with a punch of diameter $\Phi = 0.5$ mm in different positions of sections of human teeth. The imprint size allows an accurate mapping of mechanical properties but, at the same time, is great enough to avoid large scattering of data.
Furthermore, a new algorithm has been developed for analysing the experimental curves and determine the yield stress. This is quite important in view of realizing a commercial FIMEC apparatus and to carry out extensive examination of dentin and bone.

II. EXPERIMENTAL APPARATUS

During a FIMEC test, the applied load and the penetration depth are measured; it is possible to determine pressure (p) vs. penetration depth (h) curves by dividing loads by the punch-surface contact area A.
An example of FIMEC curve is shown in Fig. 2. After an initial elastic stage the typical pressure-penetration curve show three plastic stages. The first one is almost linear and ends at a pressure p_Y; in this stage the imprint shows permanent sharp edges. For $p > p_Y$ the curve slope strongly decreases (second stage) and the material starts to protrude around the imprint. Finally, the third stage shows a trend with an almost constant slope.
E is calculated from the Oliver and Pharr method [15].
Under standardised conditions (penetration rate $\cong$ 0.1 mm/min), the following correlation gives the yield stress σ_Y from the p_Y value:

$$p_Y \cong 3\sigma_Y \qquad (1)$$

Equation (1) has been verified to be valid for a lot of different materials.

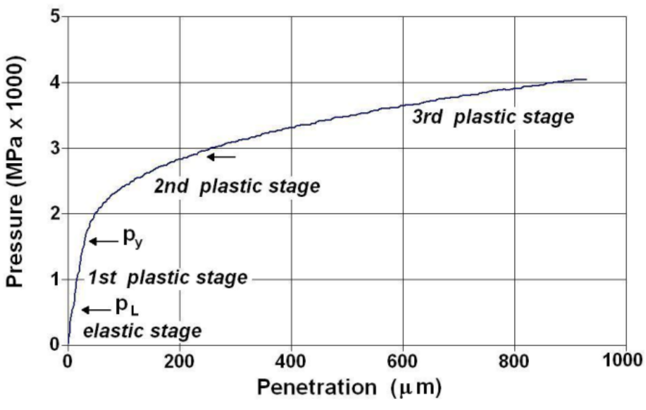


Fig. 2. Example of a typical pressure-penetration curve obtained for a steel.

An important advantage of FIMEC test with respect other indentation techniques is that the punch-sample contact area A remains constant during the test thus, elastic modulus and yield stress can be directly determined from experimental curves by applying simple analytical relationships. On the contrary, indentation with sharp or spherical punches involves a contact surface area increasing with the applied load.
The yield stress σ_Y is directly obtained from the load p_Y corresponding to the transition from the linear to the not linear stages of FIMEC curve:

$$\sigma_Y = p_Y / 3[\pi (\Phi/2)^2] \qquad (2)$$

III. DATA ANALYSIS

One of the most sensitive points for realizing a commercial FIMEC apparatus is the implementation of a suitable software for the analysis of the curves, in particular for the automatic determination of the yield stress σ_Y. After an experimental campaign carried on several materials it was possible to identify a suitable algorithm [16-17].
Owing to the inhomogeneous plastic behaviour in the initial part of punch penetration (1st plastic stage) is quite difficult to find a relationship suitable to describe this stage and useful for directly identifying the pressure p_Y for all the materials. On the contrary, the 2nd and 3rd stages, where the plastic deformation occurs in a large volume under the punch, can be described by the equation:

$$p = K (h_0 + h)^n \qquad (3)$$

where *K* and h_0 are constants, *n* the strain-hardening exponent
The following steps describe the exact procedure for the yield stress evaluation.

1- First of all the experimental pressure-penetration curve is submitted to filtering to remove possible noise, in particular the high frequency component.
2- The second step allows the evaluation of the unknown parameters. The values of *K*, h_0 and *n* are determined by the best fit of the 2nd and 3rd stages of the curve through Equation (3). This equation does not interpolate the total curve, but only the part related to the 2nd and 3rd plastic stages excluding the linear initial one. For the identification of the best-fitting curve, for the software implementation, the Ordinary Least Squared method has been used.
3- The pressure p_Y is calculated at a fixed depth

$$h_y = h_0 + h = 0.008 \qquad (4)$$

by substituting into Equation (3) the values of *K* and *n* determined by the best fitting.
The constant value $h_0 + h = 0.008$, was obtained by an iterative process of optimization carried out on the curves of several different materials. The reliability of the method has

been assessed by testing several materials of known characteristics and the scattering of data with respect those from tensile tests resulted to be within ±7%.

The Young's modulus E has been determined from the slope of the initial part of the unloading curve trough the relationship [15]:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_{eq} \sqrt{A} \tag{5}$$

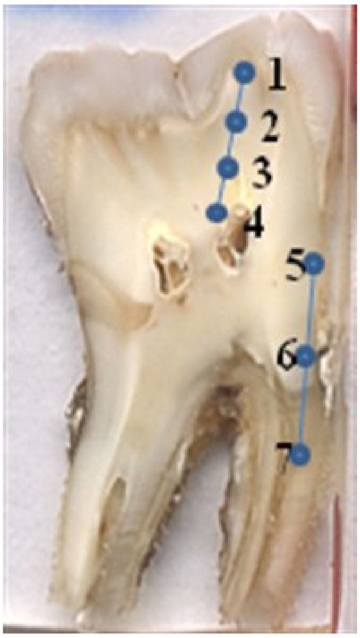
being $S = \frac{dP}{dh}$ the contact stiffness and E_{eq} the equivalent modulus defined as:

$$\frac{1}{E_{eq}} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^{\,2}}{E_i} \tag{6}$$

where E_i , E e ν_i , ν are the Young's modulus and the Poisson's ratio of indenter punch and sample, respectively.

IV. RESULTS

Results of tests carried out on dentin, in the tooth positions shown in Fig. 3 a), are presented in Fig. 3 (b-d). It is observed that yield stress and Young's modulus in different tooth positions exhibit similar trends: E decreases from the coronal dentine to the root with variations of about 20 %. The results of tests made in similar positions of different tooth sections exhibit a scattering of ± 1% and there is a good agreement with literature data obtained with various techniques [3-11].



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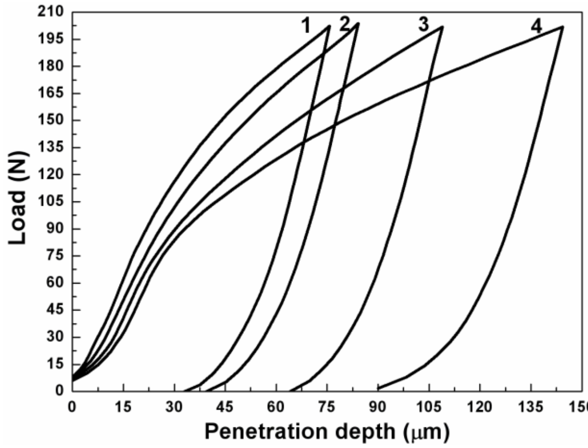

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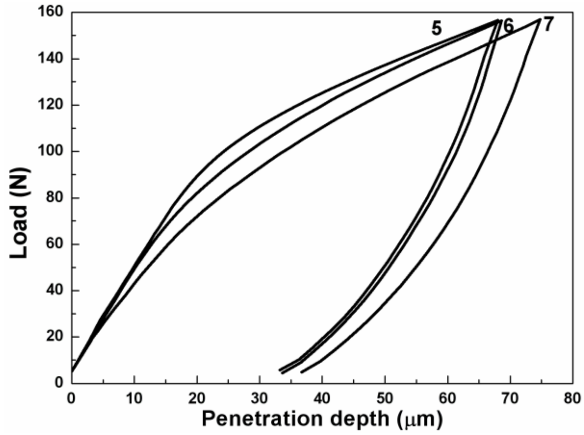

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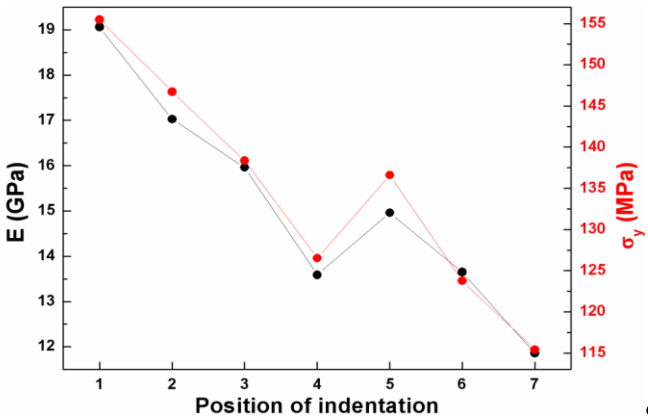

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Fig. 3. Experimental curves and trends: a) position of indentation, b) experimental results on coronal dentin; c) experimental results on radicular dentin, d) experimental trend of E and σ_Y

FIMEC tests have been exploited to measure also stress relaxation and creep.

Stress-relaxation is of particular significance in clinical situations such as use of threaded post in root canal, placement of pins during endodontic treatments and polymerization contraction of composite restorations [18-20].

To perform stress-relaxation tests, the cylindrical punch penetration has been interrupted at a load of ~ 94 N and the load evolution has been monitored for increasing time up to $4x10^3$ s, keeping constant the penetration depth. In this way load-time curves were obtained.

FIMEC stress-relaxation curves in different tooth positions are displayed in Fig. 4 a). In particular, the tests performed on positions from 1 to 3 were carried out in coronal dentin along a enamel-pulp direction, while tests on position 4 and 5 are made in two different radicular regions.

Each curve tends to an asymptotic value P_0 and can be interpolated by the function:

$$P=P_0+P_1\, e^{(-t/\tau 1)} + P_2\, e^{(-t/\tau 2)} \qquad (7)$$

To simplify, in Fig. 4 b) the P_0 term has been subtracted from the experimental data. The interpolating function consists of two exponential terms with relaxation times τ_1 and τ_2, which describe two different mechanical processes typical of porous materials.

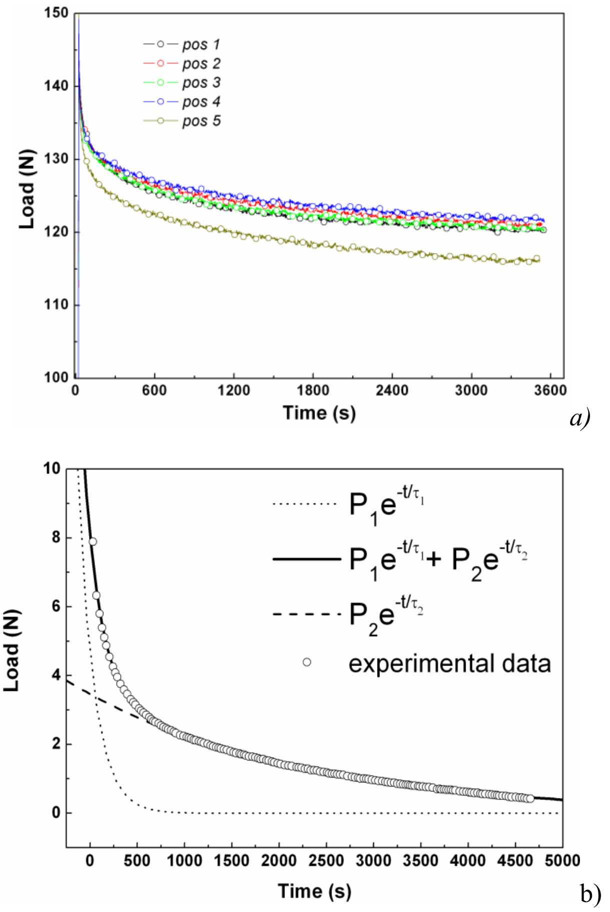

Fig. 4. Stress relaxation curves in different tooth position: 1- coronal dentin near DEJ, 2- mid coronal dentine, 3- dentine near the pulp, 4-root dentin near CEJ (cementum-enamel junction) and 5- mid root dentin; b) Interpolation of a stress-relaxation curve after subtraction of the asymptotic value P_0.

The first exponential term, $P_1e^{-t/\tau 1}$, corresponds to the initial steep load decrease due to the structural collapse of dentine walls. The second term, $P_2e^{-t/\tau 2}$, describes the real stress relaxation, i.e. the progressive and slow change of elastic into plastic strain. The values of time constants τ_1 and τ_2 depend on the position where the test is made: τ_1 varies from 67 to 90 s while τ_2 from 1200 to 1350 s.

Finally, the last mechanical properties examined in this work is creep, i.e. the dentin response under a stress constant in time. This mechanical behaviour is found in pathological situations such bruxism, i.e. the grinding of the teeth. During this event teeth are subjected to constant stress for a period ranging from some seconds to some minutes [21-22] and this event can be repeated several times, especially at night. By FIMEC test, creep values were obtained by measuring the penetration depth as a function of the time for 25 hours under a constant applied stress.

As shown in Fig. 5, the penetration rate depends on the applied stress. The slope dh/dt in the second stage (steady state creep) gives the penetration rate, being h the indentation depth and t the time.

From the curves in Fig. 5 mean values of penetration rates are 1.05×10^{-4} µm s^{-1}, 1.38×10^{-4} µm s^{-1} and 2.05×10^{-4} for the applied stresses of 56 MPa, 110 MPa and 123 MPa, respectively.

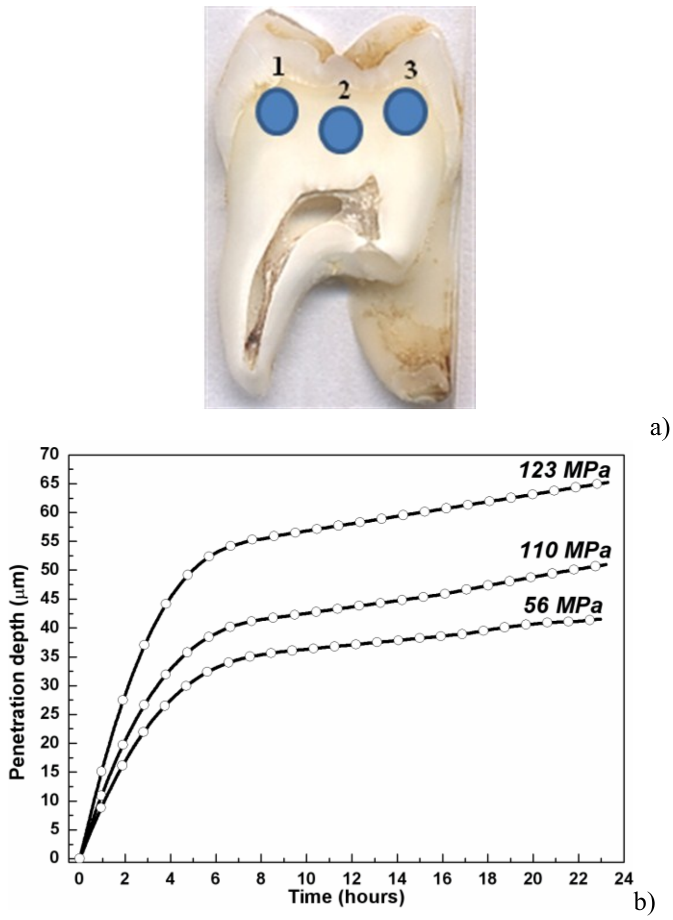

Fig. 5. Different test positions; b) Experimental creep curves: stress of 123, 110 and 56 MPa have been applied on position 1, 3, 2 respectively.

V. CONCLUSIONS

FIMEC, a technique of instrumented indentation employing a cylindrical punch, has been used to determine elastic modulus, yield stress, stress-relaxation and creep behavior in different positions of human teeth.
The main results can be summarized as follows.

1- All the values from FIMEC tests are in agreement with literature data obtained by various techniques.
2- Elastic modulus data are not affected by a so large scattering as those from micro- and nano-indentation tests.
3- Stress-relaxation curves evidenced a two-stage mechanism of deformation. They can be interpolated by a function with two exponential terms. The first exponential term corresponds to the initial steep load decrease and describes the structural collapse of the intertubular walls. The second one corresponds to the long tail of the curve and describes the progressive and slow change of elastic into plastic strain.
4- Creep curves permit to determine on a local scale penetration rate variations depending on the applied stress.

In conclusion, FIMEC proved to be a reliable methodology to measure the mechanical properties of dentin on a local scale.
A new algorithm has been developed for analysing the indentation curves in view of the realization of a commercial FIMEC apparatus.
On the basis of the results reported here, an extensive study of human teeth will be carried out in the future taking into account factors of clinical interest such as tooth type (i.e. molar, pre-molar, canine and incisive), age and gender.

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