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APLICATION OF ATOMIC FORCE MICROSCOPE FOR MECHANICAL AND TRIBOLOGICAL CHARACTERIZATION OF TEETH AND BIOMATERIALS

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Abstract: *This paper describes analysis of mechanical and tribological properties of human teeth and of their restorative materials by use of the atomic force microscope (AFM) and its combination with nanoidentation. By use of the AFM bending mode, stiffness, compliance, hardness and modulus of elasticity of dental enamel and of restorative materials are measurement. By use of the AFM lateral force the coefficients of friction of teeth and of their restorative materials are estimated, respectively. Tribological characterization of human teeth and of their restorative materials, under lubricated condition, was performing in artificial saliva which reduces the friction. Direct measurement of tribo-mechanical behavior of human teeth and of their restorative materials is of importance to clinical tooth preparation and to the development of the new artificial dental materials with a long lifetime.*

Keywords: *Teeth, Biomaterials, Hardness, Stiffness, Friction, Atomic Force Microscope.*

1. ASPECTS ABOUT THE INFLUENCE OF MECHANICAL AND TRIBOLOGICAL PROPERTIES ON THE LIFETIME OF TEETH

The surface of teeth is covered by dental enamel (Fig.1), which is the hardest tissue in the body, and it is composed of 92-96% from inorganic matter or mineral phase, 1-2% of organic material and 3-4% of water by weight [1].

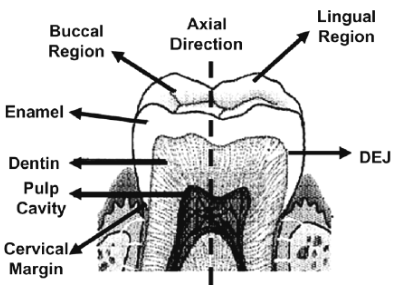


Figure 1. Schematic of tooth section [1]

Mechanical and tribological properties of enamel have a very important significance because the lifetime of tooth depends by these properties. The mechanical resistance of tooth during mastication process is influenced by hardness and modulus of elasticity of enamel. The same influence of the lifetime of tooth is given by the friction and wear of enamel during mastication.

Mechanical properties of human tooth and their restorative materials were estimated using the bending mode of atomic force microscope (AFM).

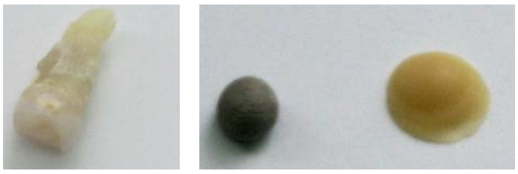
Using nanoidentation of the enamel and of dental restorative materials, hardness of surface was measured, respectively. For this an AFM cantilever with a conical tip was used.

The same works were performed of measure the modulus of elasticity, stiffness and compliance. In other, to estimate these mechanical properties a special fabricated cantilever with a spherical tip was used. The contact between sample and the spherical tip is characterized by elastically deformations of surfaced. This kind of tip was necessary to be used for obtain only elastically deformation of surface, without indentation.

Another important property to be critically considered for human tooth and their restorative materials are tribological properties. An understanding of friction and wear behavior of enamel will help the clinical treatment for increase the lifetime of human teeth. As a consequence, the AFM lateral force was used to measure the friction coefficients of enamel and of dental restorative materials under dry and lubricated conditions using artificial saliva which reduce the friction and wear.

2. MEASURMENT OF MECHANICAL PROPERTIES OF HUMAN TEETH AND OF THEIR RESTORATIVE MATERIALS

The samples for experimental tests are presented in figure 2.



a. b.

Figure 2. Sample for experimental tests: a. human tooth, b. Restorative materials: amalgam (52%Ag, 8%Cu, 0.6%Zn, 1.2%Hg) and photopolymer

Stiffness, modulus of elasticity, and hardness of samples were measured by use of atomic force microscope (AFM).

Briefly, a cantilever is used as a force sensor in AFM to detect force between tip and sample surface. The cantilever is fixed at one end and its free end has a tip, gently contacting the sample surface. A laser and a detector are used, forming an optical beam deflection system to detect the cantilever deflection. When the sample is being scanned the cantilever will move up and down in the normal direction to the surface. Of measurement the hardness of samples, the AFM cantilever is typically a silicon nitride with a conical tip. Of measurement the stiffness and modulus of elasticity, a special cantilever with a sphere as tip was used.

2.1 Stiffness of enamel of human teeth and of dental restorative materials

Stiffness is a fundamental criterion for characterization the elastically-deformable properties of a surface. This mechanical property is measurement of enamel and dental restorative materials using an atomic force microscope (AFM).

If the experimental AFM tests are performing of a very stiff surface, the displacement of piezo-table of AFM is proportional with the deflection of the AFM cantilever. In this situation there isn't deformation of surface. Of the other surface, with small stiffness the deflection of the AFM cantilever is less than the displacement of piezo-table because there is elastically deformation of surface.

The dependence between force and elastically deformation of enamel of human tooth gives the stiffness.

For estimate the stiffness of sample (without indentation), a special fabricated AFM cantilever with a sphere as tip was used (Fig.3). It is a cantilever with high stiffness, etched from highly polished beryllium bronze and coated with 300 nm thick gold layer on its top for improves the reflective properties. As tip, a steel sphere with diameter of 0.691mm was used [2].

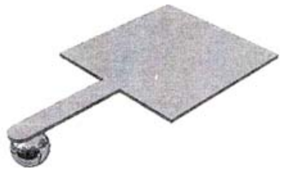
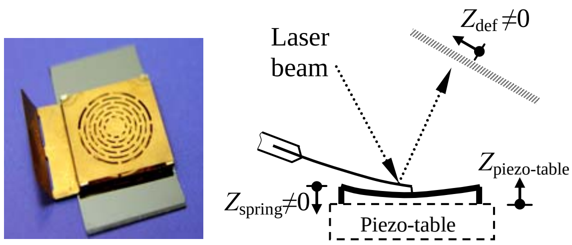


Figure 3. AFM cantilever with a sphere as tip

The stiffness of this cantilever was measurement by AFM, using a microspring with known stiffness and the following steps [3]:

- Installation into the device for the calibration a specially fabricated (etched) microspring (Fig. 4,a), made of a foil of a beryllium bronze, with known stiffness k_{spring} [N/m];



a. b.

Figure 4. Measurment of the stiffness of the cantilever with spherical tip by use a microspring: a. microspring, b. schematic of stiffness measurement

- Measurement of the bending deflection of cantilever Z_{def} [a.u.] and vertical displacement of piezo-table Z_{piezo} [nm] ;
- Correction of Z_{def} [a.u.] with a g coefficient, resulting in Z_{def} [nm]. The correction coefficient was determined of a direct contact between AFM cantilever and a hard surface (silicon wafer)

$$g = \frac{Z_{def}}{Z_{piezo}} . \qquad (1)$$

where Z_{def} is the deflection of the cantilever in arbitrary units [a.u.] and Z_{piezo} is the displacement of the piezo-table in nanometers.

- Calculation of the average value of Z_{def} [nm];
- Calculation of the average value of Z_{piezo} [nm];
- Calculation of the displacement of spring [nm]:

$$Z_{spring} = Z_{piezo} - Z_{def} . \qquad (2)$$

- Calculation of the elastic force [nN]:

$$F = k_{\text{spring}} \cdot Z_{\text{spring}} \, . \quad (3)$$

- Calculation of the stiffness of the AFM cantilever [N/m]:

$$k_{\text{cantilever}} = \frac{F}{Z_{\text{def}}} \, . \quad (4)$$

The stiffness of the cantilever with spherical tip, used in this experiment was determined of 220N/m.

During experimental tests, the deflection of the cantilever is measurement. This deflection and the stiffness of cantilever give the force.

$$F = k_{\text{cantilever}} \cdot Z_{\text{def}} \, . \quad (5)$$

Deflection of the AFM cantilever Z_{def} as function of the displacement of piezo-table Z_{piezo} is determined for enamel of human tooth and for other dental restorative materials. Of these measurements the same AFM cantilever with known stiffness is used.

The difference between displacement of piezo table and deflection of AFM cantilever gives the elastically deformation of surface of sample:

$$Z_{sample} = Z_{\text{piezo}} - Z_{\text{def}} \, . \quad (6)$$

By use of the maximum elastically deformation of samples and the maximum force gives by the cantilever, the stiffnesses of samples can be calculated as:

$$k_{sample} = \frac{F}{Z_{sample}} \, . \quad (7)$$

Table 1 presents the results of measurement of stiffness and compliance of human tooth and of dental restorative materials.

Table 1. Stiffness of enamel of human teeth and of other dental restorative materials

Samples	Stiffness [N/m]	Compliance [m/N]
Human tooth	1103	0.0009
Amalgam	166.58	0.006
Photopolymer	96.76	0.01

2.2 Modulus of elasticity of enamel of human teeth and of dental restorative materials.

In order to measure the modulus of elasticity of human tooth and of their restorative materials, the following equation is used [2]:

$$E = \frac{3}{4}\left(1 - \nu^2\right)\frac{k}{r^{1/2}} \cdot \frac{Z_{def}}{\left(Z_{piezo} - Z_{def}\right)^{3/2}} \, . \quad (8)$$

where ν is the Poisson's ratio of materials, k – the stiffness of the AFM cantilever, Z_{piezo} – position of the piezo-table of the AFM, Z_{def} – deflection of the AFM cantilever, r – radius of the sphere of the cantilever.

By use a cantilever with 0.345mm the radius of sphere and the measurement Z_{piezo} and Z_{def}, the elastically deformation of surface is determined by use equation (6) and after than the modulus of elasticity using equation (8). The results of these measurements of enamel and other dental restorative materials are presented in Table 2.

Table 2. Modulus of elasticity of enamel of human teeth and of other dental restorative materials

Samples	Deformation of surface [nm]	Modulus of elasticity [GPa]
Human tooth	231.6	113.5
Amalgam	793.1	96.6
Photopolymer	967.9	25

2.3 Hardness of enamel of human teeth and of dental restorative materials.

In other to measure the hardness of enamel and of dental restorative materials, an AFM cantilever with conical tip was used. A nanoidentation of surfaces of samples were performed using this AFM cantilever. The height of the deep of nanoidentation depends by the hardness of surface and it can be calculated as the difference between displacement of piezo-table and deflection of the AFM cantilever.

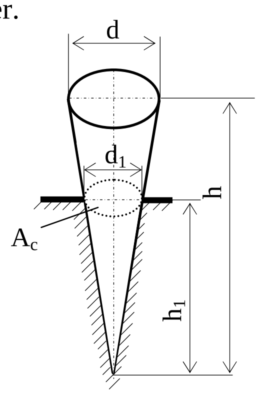


Figure 5. Contact area A_c gives by the indentation of sample with an AFM conical cantilever

The area A_c of sample (Fig.5), results after indentation with the conical cantilever, can be calculated as:

$$A_c = \frac{\pi}{4}\frac{d^2 h_1^2}{h^2} \, . \quad (9)$$

where d is the diameter of the tip, h_1 - the height of the indentation deep, and h- the height of the tip of the AFM cantilever.

The indentation force of the AFM cantilever can be calculated as:

$$F = k_{cantilever} \cdot Z_{def} . \qquad (10)$$

where the stiffness of the AFM cantilever $k_{cantilever}$ is known, and Z_{def} is given by the AFM.

Hardness H of sample is equal to the force F divided by the contact area A_c, when the indenter is at full load. Using equations (9) and (10), the hardness of teeth and ot their restorative materials can be calculated as:

$$H = \frac{F}{A_c} . \qquad (11)$$

Table 3 shows the hardness of enamel of human tooth and of other dental restorative materials.

Table 3. Hardness of enamel of human teeth and of other dental restorative materials

Samples	Height of indentation deep [nm]	Hardness [GPa]
Human tooth	45	5.68
Amalgam	360	3.67
Photopolymer	740	1.26

3. MEASURMENT OF FRICTION COEFICIENTS OF HUMAN TEETH AND OF THEIR RESTORATIVE MATERIALS

The tribological experiments were performed by use of the AFM lateral mode.

The AFM cantilever if moving in lateral direction on sample and the rotational deflection of the AFM cantilever is measurement. This rotational deflection of the AFM cantilever is proportional with the friction coefficient. The material of AFM cantilever is Si_3N_4. The fretting tests were performed under dry condition and under lubrication, using artificial saliva.

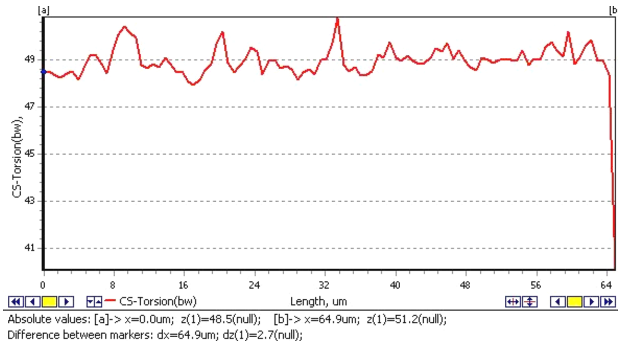


Figure 6. Rotational deflection of the AFM cantilever of enamel under dry condition

By use the deflection (dz) of AFM cantilever gives by the torsion map (Fig.6) the friction force can be calculated as:

$$F_f = \frac{dz \cdot r \cdot G \cdot h^3 \cdot b}{l^2 \cdot s} . \qquad (12)$$

where dz is the deflection of AFM cantilever [nm], r – the radius of tip of AFM cantilever, G – shear modulus of the material of the AFM cantilever, l – length of AFM cantilever, t – thickness of AFM cantilever, b – width of AFM cantilever, s - height of tip of AFM cantilever.

The friction coefficient can be calculated by use the friction force and the normal force gives by the stiffness of the AFM cantilever (k_{AFM}) and vertical deflection of it (Z_{AFM}).

$$\mu = \frac{F_f}{k_{AFM} \cdot Z_{AFM}} . \qquad (13)$$

Table 4 presents the friction coefficients of human tooth and of other dental restorative material under dry friction and under lubricated conditions.

Table 4. Coefficients of friction of enamel of human teeth and of other dental restorative materials

Samples	Coefficient of friction	
	Dry condition	Lubricated condition
Human tooth	0.18	0.08
Amalgam	0.23	0.14
Photopolymer	0.52	0.19

The friction coefficients of samples are lower to 0.2 under lubricated conditions (artificial saliva), compared to dry conditions where the friction coefficient is from 0.18 to 0.52.

The tooth analyzed of these experiments was from a man which is 51 years old. Of course, mechanical and tribological properties of human teeth depend strongly upon individual difference (age, physical conditions).

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