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THE INFLUENCE OF TEMPERATURE ON MECHANICAL AND TRIBOLOGICAL PROPERTIES OF DENTAL MATERIALS

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ABSTRACT

The dental enamel is the hardest surface of teeth. The lifetime of teeth depends by mechanical and tribological properties of enamel. These properties are influenced by the mastication conditions including humidity, temperature and abrasion effects. The scope of this paper is to analyze the temperature effect on tribological and mechanical properties of restorative dental materials using advance techniques. Nanoindentation provides information about hardness and elastic modulus. The tribological investigation in this paper is resumed to friction force measurement as a function of temperature. A temperature control system is used to monitor the temperature of investigated samples in the range of 10˚C to 60˚C. An atomic force microscope with a nanoindentation module is used to determine the mechanical and tribological properties of dental materials as a function of temperature. These relatively nondestructive mechanical characterization techniques may assist in better understanding the mechanical behavior of the dental materials and thus facilitate their preparation with excellent mechanical and tribological properties.

Keywords: Dental materials, nanoindentation, thermal effect, hardness, friction.

INTRODUCTION

The human teeth are the hard, resistant structures occurring on the jaws and in around the mouth area of vertebrates. The teeth are used, in principals, for masticating food, and for other specialized purposes. A tooth consists of a crown and one or more roots. The crown is the functional part that is visible above the gum. The root is the part that cannot see and supports and fastens the tooth in the jawbone. The shape of the crown and root vary among different teeth in the human mouth.

Hardness is the mean pressure that a material bears under load. This parameter is experimentally affected by several geometrical uncertainties, such as penetration depth, size and shape of the indenter.

All teeth have the same general structure and consist of three layers (Lewis and Dwyer-Joyce, 2005) as shown in Fig.1. The hardest tissue in the body is an outer layer of enamel, which is wholly inorganic. The enamel covers part or the entire crown of the tooth. The typical value for enamel hardness range reported in literature is from 1 GPa to 8 GPa. Regarding enamel elastic modulus, we can say that the values range from 19.9 GPa to 91 GPa when the measurements are perpendicular to crystal orientation, and from 93 GPa to 113 GPa when the measurements are parallel to crystal orientation (Srivicharnkul et al., 2005).

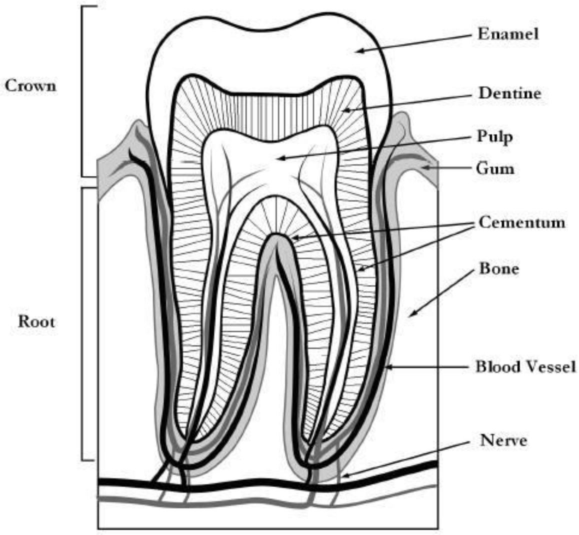


Fig. 1 Human tooth section (Lewis and Dwyer-Joyce, 2005)

Anatomically speaking, the crowns of teeth are covered by dental enamel, which consists of 92% − 96% inorganic matter, 1% − 2% organic material and 3% − 4% water by its weight (Gwinnett, 1992).

Enamel hardness is attributed to its high mineral content (Caldwell et al., 1957) and on the other hand the brittle property is due to its high elastic modulus and low tensile strength (Meckel et al., 1965).

Despite enamel being a very strong substrate, clinically, enamel cracks and fractures can occur. Studies have shown that enamel is anisotropic material and its mechanical properties may be dependent on the type and direction of the stress applied, as well as the prismatic orientation (Urabe et al., 2000; Rasmussen et al., 1976; Hassan et al., 1981; Xu et al., 1998).

The main reasons for placing a dental restoration are: primary caries or noncarious defect such as abrasion/erosion, traumatic tooth fracture, developmental defect, cosmetic reasons, and restoration of an endodontically treated tooth or other unspecified defects or reasons (Giannini et al., 2004).The destruction of healthy tissue has always been a big concern, and still today, the caries represent the most widespread human disease (Langeland, 1987).

Therefore restorative materials are expected to replace and perform as natural tooth materials. The demand of achievement is so great that most of the times restorative filling materials replace enamel and dentin, which have very different mechanical properties, namely hardness and elastic modulus. Thus, the goal of research when developing these restorative materials is to develop the ideal restorative material which would be identical to natural tooth structure, in strength adherence and appearance.

Mechanical and tribological properties of direct restorative filling materials are crucial not only to serve and allow similarity with human enamel and dentine but also to compare composites between them and determine objective criteria for their selection.

The objective of this work is to investigate the hardness and friction behavior of same commercial restorative materials using the nanoindentation and the AFM techniques.

MATERIALS. PREPARATION OF THE SAMPLES

Four commercial dental composite resins were investigated in this work. All tested composites are one-paste systems (Fig. 2). Information regarding their classification, indication, monomer composition, type and size of reinforcing filler particles, selected shade and the manufacturer are summarized in the following table. Data were provided by the samples manufacturer.

(http://www.kerrdental.com/kerrdental-msds-us-english).

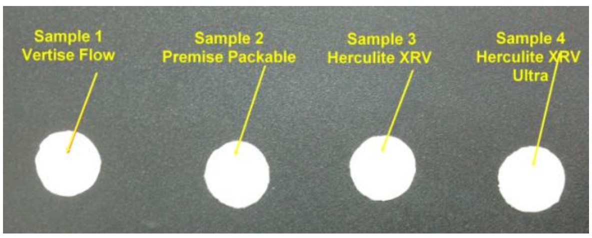


Fig. 2 Samples for experimental tests

Table 1* Classification of resin composites regarding filler size and size distribution (clinical application)

Type of composite resins/ commercial name	Samples dimensions (disc)	Particles size. characteristics	Clinical use
XRV Herculite Ultra Nanohibride resin	Diameter - 15mm Width – 1mm Hand made Curing time: 40 seconds.	Enamel type, color A1, reinforcement particles which range from ~ 0:5 μm to 3 μm.	Moderate stress areas requiring optimal polishability
XRV Herculite Microhibride resin	Diameter - 15mm Width – 1mm Hand made Curing time: 40 seconds.	Enamel type, color A2 large filler particles, with an average size of 15 μm − 20 μm and also a small percentage in weight of colloidal silica, which has a particle size ranging from 0:01 μm m to 0:05 μm m.	High-stress areas requiring improvement polishability
Premise Packable Trimodal hybride resin,	Diameter - 15mm Width – 1mm Hand made Curing time: 40 seconds.	Dentine type, color A3, Universal Trimodal Nanocomposite featuring a unique 3-filler blend (0.02 μm; 0.4 μm and Pre-polymerised filler) and 84% filler loading, Midifiller/minifiller hybrid, but with lower filler fraction	Situation in which improved condensability is needed
Vertise Flow Uncured methacrylate ester monomers	Diameter - 15mm Width – 1mm Hand made Curing time: 40 seconds.	Midifller hybrid, but with fine particle size distribution Non-hazardous inert mineral fillers, non-hazardous activators and stabilizers	Situation in which improved flow is needed and/or where access is dificult

****Observation:*** All samples were prepared in the Laboratory of Faculty of Dental Medicine, from Cluj-Napoca, Romania.

NANOINDENTATION TESTS

Knowledge of the mechanical properties of the dental materials is crucial for understanding how masticators strains are distributed throughout a tooth, and for predicting how stresses and strains are altered by dental restorative procedures, age and disease (Kinney et al., 1996). It is expected that under mastication loadings, a restoration with sufficient and identical mechanical properties to that of the adjacent tooth structure will have a longer lifetime (Angker and Swaina, 2006).

Recently, indentation tests are becoming most commonly applied means of testing the mechanical properties. An atomic force microscope XE 70 (manufactured by the Park System Company) with a nanoindentation module enable the measurement of hardness on the surface of dental materials is used in experiment. A temperature control system based on Peltier element is used to monitor the temperature of samples in the range from 10°C to 60°C. All experiments were performed in a clean room with control of humidity, temperature and air pressure.

Indentation tests are the most used way of testing the hardness of materials. This technique has its origins in the Mohs scale of mineral hardness and has been extended in order to evaluate material hardness over a continuous range. Hence, the adoption of the Meyer, Knoop, Brinell, Rockwell and Vickers hardness tests were performed. The nanoidentation technique has been established as the primary tool for hardness investigations of micro/nano scale. The test is usually performed with a pyramidal or a conical indenter.

Indentation data were obtained of investigated dental materials using an indentation force of 250µN. The tests were repeated three times. A Berkovich diamond tip (three-sided pyramidal) was used for all indentations. Temperature of investigated material was modified during testing from 10ºC to 60ºC.

The analysis method used in the experimental determination of hardness is the Oliver and Pharr method. This is a standard procedure for determining the hardness and elastic modulus from the indentation load-displacement curves at micro and nano - scale. Hardness for each material was determined from the load-displacement curves during unloading. The Oliver-Pharr method was proven to be an efficient tool for mechanical characterization of soft or hard materials (Oliver, 1992; Angker and Swaina, 2006; Drummond, 2006).

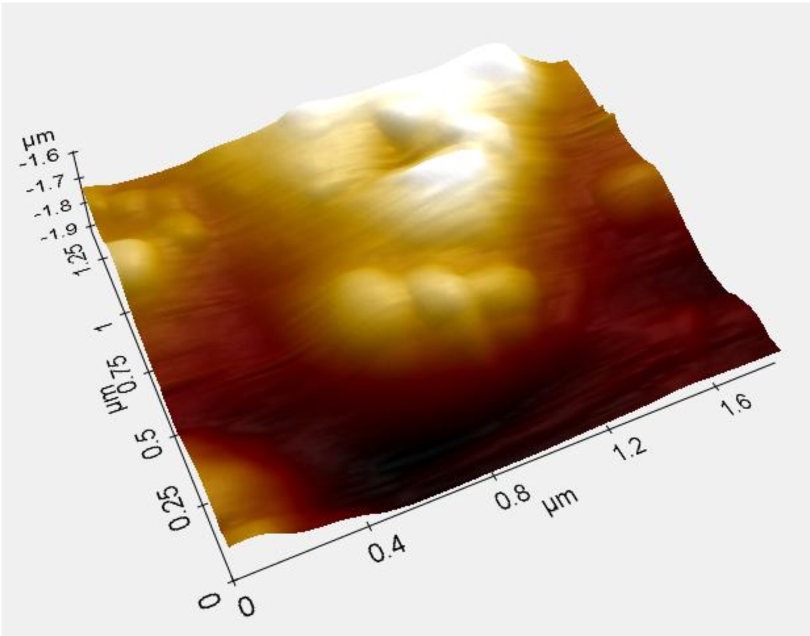


Fig. 3 Nanoindentation of Vertise Flow dental material at 20ºC with a force of 250µN

Before and after indentation process at each temperature, the AFM contact mode was used to scan the surface. The topography of the samples surface was then obtained by AFM scanning. Figure 3 shows the nanoindentation place of the Vertise Flow material at 20ºC with an indentation force of 250 µN. The trace of a series of indentation on the Vertise Flow material at different temperatures is shown in Fig.4. The largest trace represents the indentation carried out at 60ºC while the smallest trace represents the indentation at the 10ºC. The optical microscope of the AFM test system was used to accurately locate the regions of interest. Indentations were spaced sufficiently far apart so that the indentation behavior was not affected by the presence of adjacent indentations. The instrument's software corrected all data for thermal drift and instrument compliance.

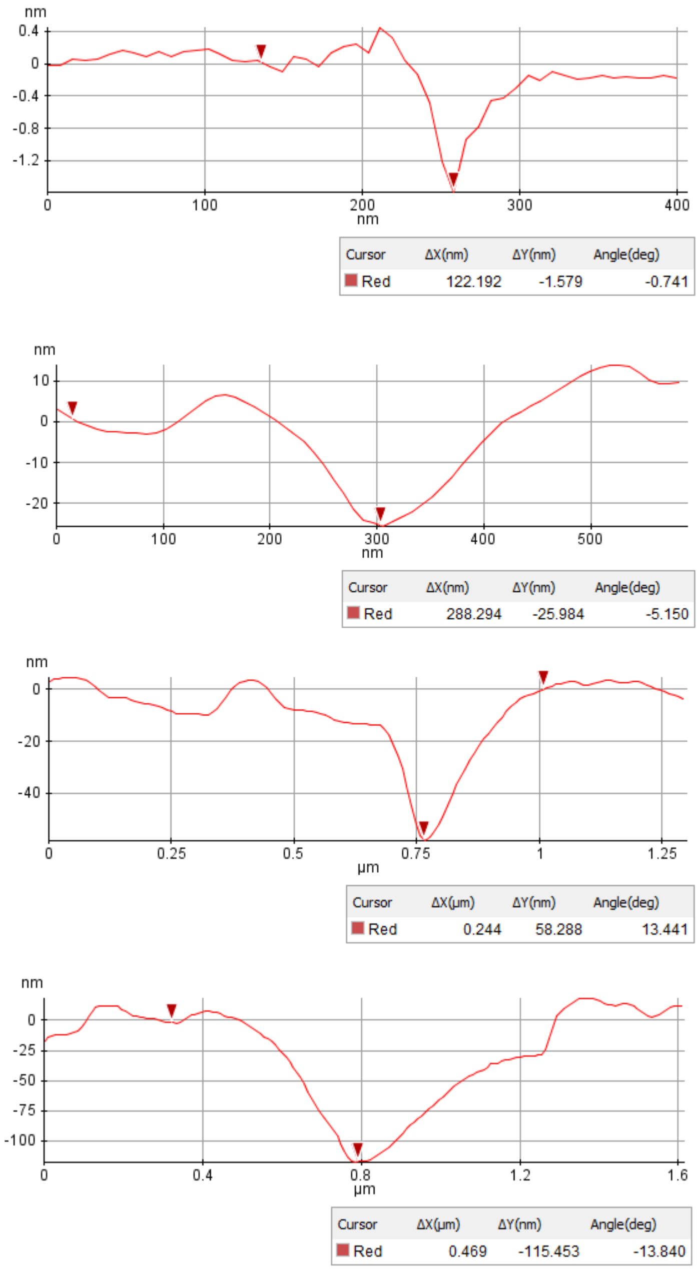


Fig. 4 Indentation depths of Vertise Flow material at different temperature for a force of 250µN

Using the XEI Software the cross-section of the indentation places are obtained in order to measure the indentation depth. Figure 4 shows the variation of the maximum indentation depth as a function of temperature for a force equal by 250 µN. As temperature increases the indentation depth increases respectively. The same procedure is used for all the investigated samples. The results of the variation indentation depth as a function of temperature are presented in Fig. 5.

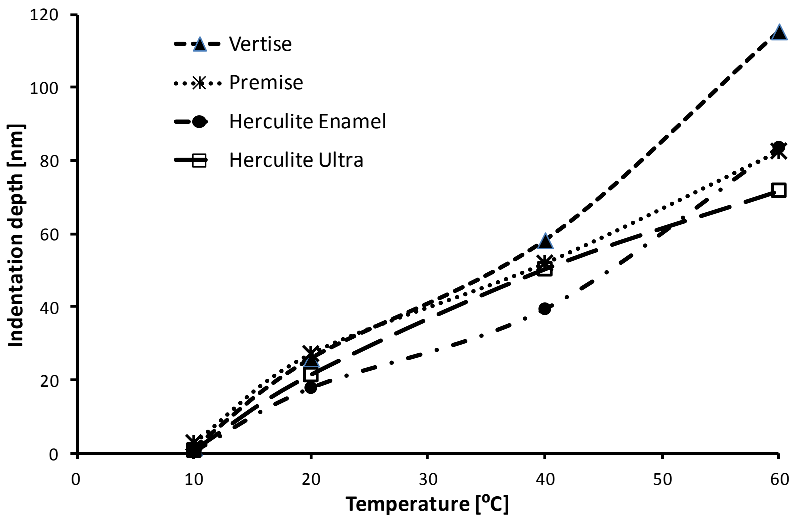
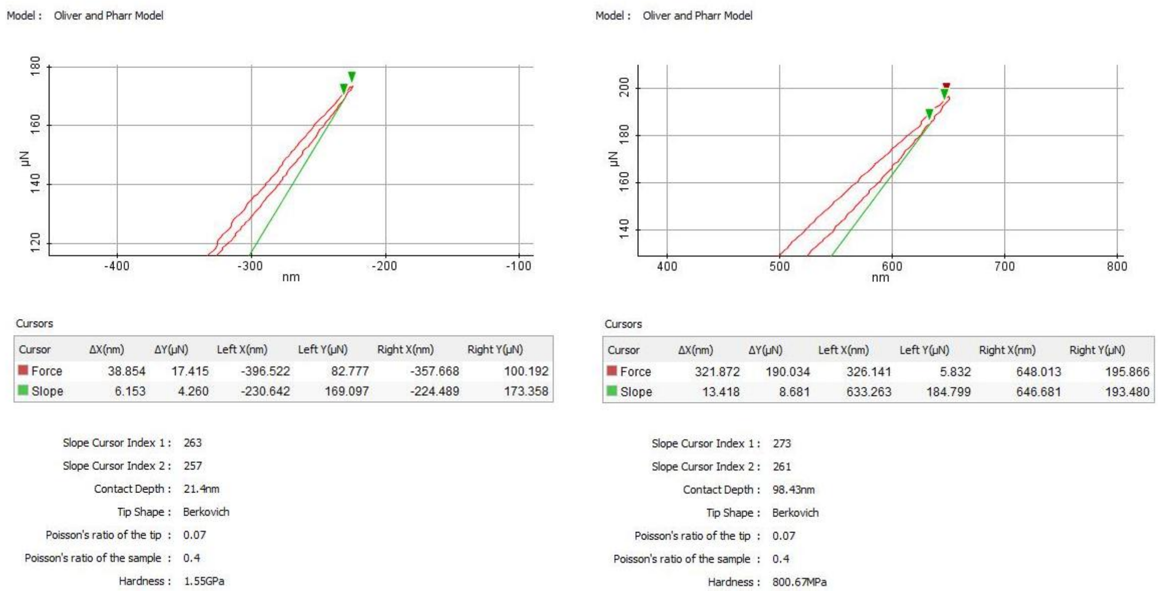


Fig. 5 Indentation depths variation as a function of temperature of investigated dental materials

As temperature increases the indentation depth increases, respectively, under the same indentation force. As a consequence, the surface contact stiffness is changed and the hardness is modified.



(a) (b)

Fig. 6 Hardness variation as a function of temperature of Vertise Flow dental material at: (a) 20°C and (b) 60°C

The hardness were estimated by using XEI Software based on the Oliver-Pharr approach, analyzing the load-unload curves performed during experimental campaign. Hardness is directly provided by software. Figure 6,a presents the hardness of Vertise Flow material at 20°C, which is equal by 1.55GPa for a contact depth of 21.4nm. As temperature increases of 60°C, the hardness decreases, respectively. For the same material, using the same indentation force, a hardness of 0.8GPa is experimentally obtained at 60°C for a contact depth of 98.43nm (Fig.6b).

The same experiment was performed for all investigated dental materials for different temperatures. The results are presented in Table 2.

Table 2 Hardness (GPa) of investigated dental materials for different temperatures

Material	Temperature [^oC] 10^oC	20 ^oC	40^oC	60^oC
	Hardness			
Vertise Flow	1.7	1.55	1.27	0.8
Premise Packable	2.82	2.6	1.91	1.15
XRV Herculite	1.87	1.62	1.38	0.95
XRV Herculite Ultra	2.59	2.2	1.81	1.24

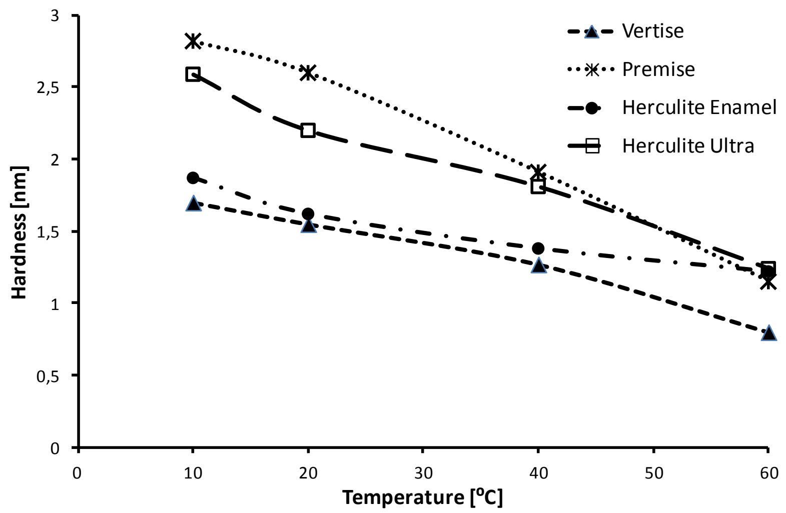


Fig. 7 Hardness variation as a function of temperature of investigated dental materials

The obtained results from the nanoindentation tests confirm hardness value in the range of 1 to 3GPa of investigated dental material at the room temperature. The hardness decreases as temperature increases. The theoretical interpolation of the hardness variation as a function of temperature is presented in Fig.7. The measured mechanical properties of dental resins at ambient temperatures are in the same range of those reported with other studies taken from literature (Angker and Swaina, 2006).

TRIBOLOGICAL TESTS

When the intent is to study wear behavior between dental materials that is strongly influenced by friction it is necessary to understand the environment in which they work, as well as the mechanisms involved in the processes which are the most important and finally conceive and/or use equipment adapted to the aim of the study.

The energy dissipated by friction between the bodies in contact can be considered the major energy source of wear of materials in sliding contacts. The Coulomb friction model establishes that the friction force is proportional to the normal load, therefore, assuming a constant friction coefficient, a proportional relationship can be established between the wear volume and friction force. From the energetic approach the energy dissipation is also directly proportional to the wear volume (Hamilton, 1983; Johnson, 1985).

During the chewing process of human beings, the magnitude of mastigatory force in the oral cavity ranges from 3 N to 36 N (Dowson, 1998; Goldmann and Himmlova, 2008). In order to observed the wear of teeth in its incipient phase the micro and nano tests are needed. In our tests, the temperature influence a friction force where a normal load of 10µN is investigated using the lateral mode of AFM.

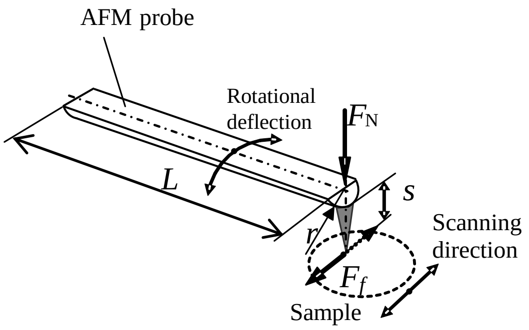


Fig. 8 Scanning principle and geometrical dimensions of an AFM probe

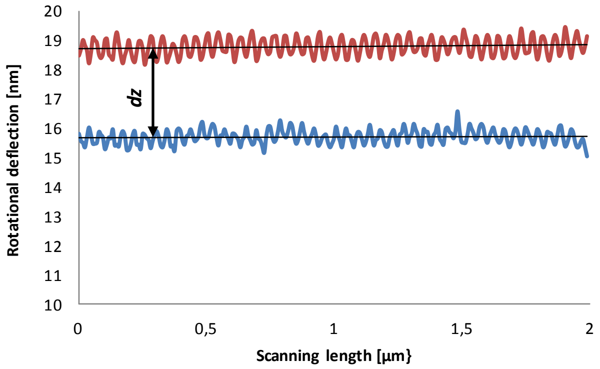


Fig. 9 Rotational deflection of AFM cantilever during sliding (contact - Diamond on Premise Packable material)

The friction is estimated by AFM measurements of the rotation deflection of AFM probe. In that case, the two surfaces in contact are the tip of AFM probe and the sample. This measurement provides an index of friction behavior between two materials being in contact and in relative motion. The relative motion between tip and surface is realized by a scanner composed of piezoelectric elements, which move the material surface perpendicular to the tip of the AFM probe with a certain periodicity as shown in Fig. 8. The scanner can also be extended or retracted in order to modify the normal force applied to the surface. This force gives information on the rotational deflection *dz* of AFM probe (Fig.9). The relative sliding of the AFM probe tip on the top surface of investigated materials is influenced by friction. The lateral force, which acts in the opposite direction of the scan velocity, causes torsion of the AFM probe. Using a photo-detector the lateral movements of the AFM probe during scanning is determined.

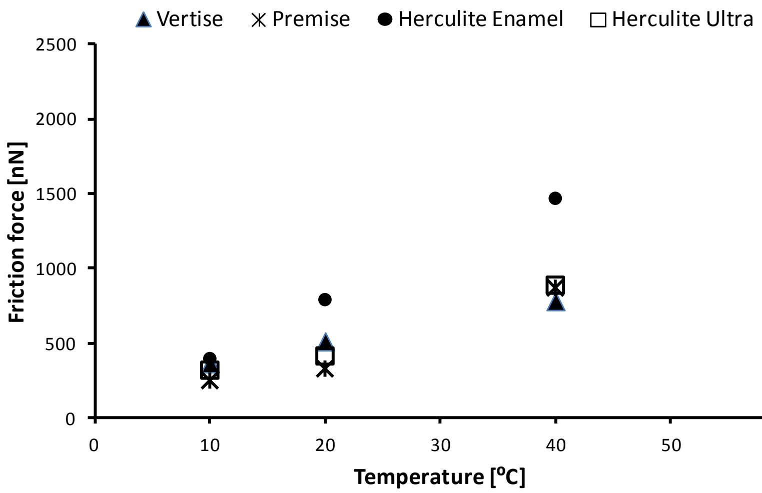


Fig. 10 Temperature influence on friction forces between the AFM tip (diamond) and investigated dental materials

In order to avoid the temperature influence on the AFM cantilever during scanning, a probe with high stiffness (144N/m) and with a diamond tip is used. The AFM probe has the following geometrical dimensions (Fig.8): tip height s = 109μm; radius r = 24μm; length L=782 μm.

Rotational deflection *dz* of AFM probe is measured (Fig.9) and the friction force is evaluated with the following formula that was computed based on the torsion beam theory:

$$F_f = \frac{0.3r^4 \cdot G}{L^2 \cdot s} \cdot dz \tag{1}$$

where *dz* is the calibrated deflection of AFM probe [nm]; *G* – is shear modulus of the cantilever material; *L, s, r* – are the dimensions of AFM probe.

Using the same normal load of 10μN for all samples, the variation of the friction forces as a function of temperature is determined. The results are presented in Fig. 10.
The rotational deflection of AFM cantilever increases if the temperature increases, respectively. If the friction forces of all investigated dental materials are in the same range at the room temperature, as temperature increases, the materials have different behaviors, especially for XRV Herculite Enamel.

CONCLUSIONS

Investigations of the mechanical and tribological properties at micro/nano-scale using the atomic force microscope can provide insights into failure mechanism of dental material. In this paper, the temperature influence on hardness and friction is investigated. Nanoindentation is an attractive method for measuring the mechanical behavior of small specimen volumes in dental hard materials. Using this technique, the mechanical properties of nanocomposite resins were investigated. Experimental study has been carried out at different temperatures representing several steps of severity conditions for materials in test.

This study has enabled to conclude that, the hardness of dental materials decreases as temperature increases. At room temperature, Premise Packable and XRV Herculite Ultra materials have high hardness. Tribological investigation consider friction force measurement that is evaluated based on the rotational deflection of AFM probe. A high value of friction force is determined for XRV Herculite Enamel. As temperature increases the friction force increases because the surface contact stiffness decreases that made the AFM probe sliding heavier on investigated materials.

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