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ULTIMATE STRENGTH OF DENTAL NANOMATERIALS UNDER STATIC LOADING

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ABSTRACT

Knowing the engineering behavior of dental materials isof great importance,because it enables the proper material selection and decision for the long-time clinical success. Exposure of these materials to mechanical tests in order to determine their resistance provides information which can serve as guidelines for clinical practice. Nanocompositesare the most commonlyused nanomaterials in contemporary dental practice. It is expected that these materials are strengthened by the presence of nanoparticles. The aim of this study was to determine mechanical properties of contemporary dental resin-based nanocomposites.
Key words: *Mechanical properties, Compressive test, Diametral test, Vickers hardness test, Dental nanocomposites*

1. INTRODUCTION

Clinical success of dental treatment does not depend only on knowledge of biological, physiological and pathophysiological principles, but also on a complete understanding of the fundamental properties of biomaterials used in restorative purposes[1]. Dental operator has to be familiar with the engineering aspects of dental materials and to be able to make a proper selection of materials in a specific clinical situation [2]. Only integrative knowledge can provide the best possible prognosis of each individual restorative treatment. Mechanical properties of dental materials are one of the essential material properties that can affect the dental clinical success [3]. Modern dental treatment methods extend the lifetime of teeth, causing more frequent need for high quality, resistant materials for long-term and stable therapeutic results. Considerable effort is invested in research and improving the overall dental materials properties, including their mechanical behavior.

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Dental materials have to be sufficiently mechanically resistant to withstand static and dynamic occlusal forces that are produced during chewing [4]. These materials should adequately serve the function for a longer period of time in complex conditions of the stomatolognatic system [5,6].
The most commonly used restorative materials in dental practice are resin-based composites [7]. These materials are tooth-colored materials that consist of organic matrix, inorganic fillers and organo-silane – bond between these two constituents [8,9]. Dental composites are photo-polimerizable materials, whose polymerization starts when the material is exposed to visible light energy source [8,9]. Composite materials can be classified according to different criteria for division. One of the most frequently used criteria for division is dimension of inorganic filler particles. Dental composites can be divided into: macrofilled, microfilled, microhybrid, nanofilled and nanohybrid materials, considering dimensions of inorganic particles (fig. 1) [10]. The effect of composite composition on the material properties is a well-known phenomenon. Contemporary dental nanocomposites were created in order to overcome the shortcomings of previous materials and to improve the overall material characteristics [8,9]. Nanoparticles have unique phisicochemical properties and high surface area, and because of that they have the ability to strengthen the material [11,12]. Further, high inorganic volume fraction reduces the proportion of organic matrix responsible for the negative material features, such as: high polymerization contraction, high coefficient of thermal expansion, sorption and solubility, low mechanical strength, low wear resistance etc[8,11,13,14,15,16]. Generally, increased volume fraction of fillers improves better quality of composite material. According toDe Souza et al. the most common reason for dental composite failure, during function in oral environment, is polymerization shrinkage, and the second one is deficient mechanical strength and the fracture [3].

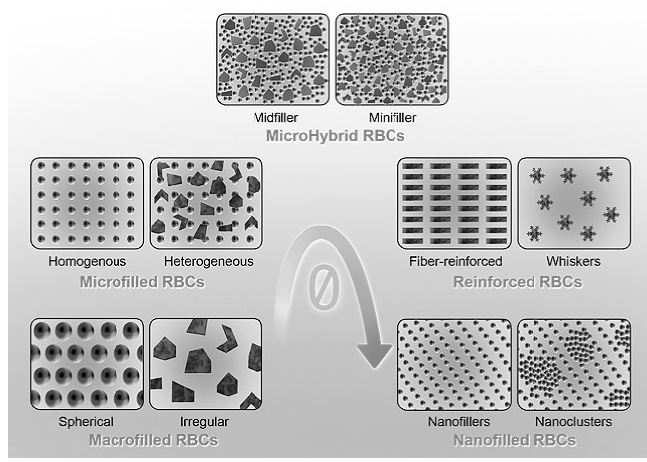


Fig.1- *Diagrammatic summarization of development of filler particles in resin-based composites (RBCs*) [17]

Dental nanocomposites are expected to be the materials with great optical and aesthetic characteristics, as the microfilled composites, which are used for the restoration of frontal teeth. Also, it is expected that nanocomposites have very good mechanical strength, close like or even higher than microhybrid composites, usually recommended for the use in lateral occlusal region [18].

Contemporary dental resin-based nanocomposites are divided into two main groups of materials: nanohybrid and nanofilled materials [19]. Nanohybrid composites consist of particles of different sizes, some in micrometer and some in nanometer dimensions (eg 2 μm diameter particles mixed with particles of less than 50 nm in diameter). Nanofilled composites contain particles of more uniform size range, all below the "nano" limit of 100 nm (eg, a combination of particles which are 75 nm in diameter with particles of 5-25 nm in diameter) [8,10,15].
The aim of this study was to determine mechanical properties of contemporary dental resin-based nanocomposites and to compare these properties with universal restorative microhybrid composite, used as a reference material.

2. MATERIALS AND METHODS

Three representative dental resin-based composites were tested in the study: nanofilled (Filtek Ultimate Body, 3M ESPE), nanohybrid (Filtek Z550, 3M ESPE) and microhybrid (Filtek Z250, 3M ESPE). Detailed informations about materials used in the study are shown in the table 1.
Specimens of each material were made by using cylindrical molds made by Rapid prototyping technology. Dimensions of specimens were ϕ4x4mm for compressive test, ϕ5x2mm for diametral tensile test and ϕ4x2mm for Vickers hardness test. Molds were placed on the glass microscope slide, filled with material and covered with another glass slide, taking care to obtain a flat surface without any defects and entrapped air. Material was then polymerized for 40 seconds with a SmartLite® IQTM 2 LED unit (Dentsply Caulk).
After specimen preparation experimental tests were conducted. Mechanical press with 50kN rated force was used for compressive test and diametral tensile test. For force and stroke measurement force transducer, displacement transducer and Spider 8 universal amplifier were used. Experiments were conducted in Laboratory for Materials on Faculty of Technical Science. Vickers hardness testing machine was applied for the hardness test.
Schemes of the applied tests are presentedin figure 2. Hardness was measured on both sides of the specimen (TOP and BOTTOM) using diamond indenter in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces (figure 2a), subjected to a load of 98,1N. Loading time was 30s. Three specimens of each material were used, as it is shown in figure 3a. In this test, specimens were remained in the moulds due to easier positioning on the test machine.Six measurements per specimen were carried out, three on top and three on bottom side of each specimen.After indentation process diagonals of imprint were measured (figure 4a). For calculation of Vickers hardness HV equation (1) was used:

$$HV = 0{,}1891 \cdot \frac{F_V}{d^2} \tag{1}$$

where:

F_V =98,1N – force applied in process [N]
$d=(d_1+d_2)/2$ – average value of diagonals [mm]

For compressive test 3 specimens of each material with ϕ4x4mm dimensions were used. Procedure was carried out as shown on figure 2b. One of the used specimens before test is presented in figure 3b. Specimens were compressed with flat dies. Press velocity was 10mm/min. No lubrication was applied. Process was conducted until the crack. Values of compressive strength *p*were calculated according to equation (2).

$$p = \frac{F_{CT}}{A^2} = \frac{4 \cdot F_{CT}}{D_{CT}^2 \cdot \pi} \left[\frac{N}{mm^2} \right] \tag{2}$$

where:

F_{CT} - force occurred in experiment [N]
D_{CT} =4mm - diameter of specimens [mm]

Table 1. *Details of the materials tested in the study**

Material	Filtek Ultimate Body	Filtek Z550	Filtek Z250
Manufacturer	3M ESPE, St. Paul, MN, USA	3M ESPE, St. Paul, MN, USA	3M ESPE, St. Paul, MN, USA
Classification	Nanofilled	Nanohybrid	Microhybrid, St Paul, MN, USA
Lot no.	N349776	N340139	N367949
Shade	A2	A2	A2
Matrix	Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA	Bis-GMA, UDMA, Bis-EMA, TEGMA and PEGDMA	Bis-GMA, UDMA, Bis-EMA, TEGMA
Fillers	Non- agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4-11 nm zirconia filler, and aggregated zirconia/silica cluster filler (average cluster particle size – 0,6-10 μm)	Surface-modified zirconia/silica fillers 3000 nm (3 μm or less), non-agglomerated/non-aggregated surface-modified silica particles 20 nm	Zirconia, silica 10 – 3500 nm (0,01-3,5 μm)
Filler loading	78,5 wt%, 63,3 vol%	82 wt% 68 vol%	75-85 wt% 60 vol%

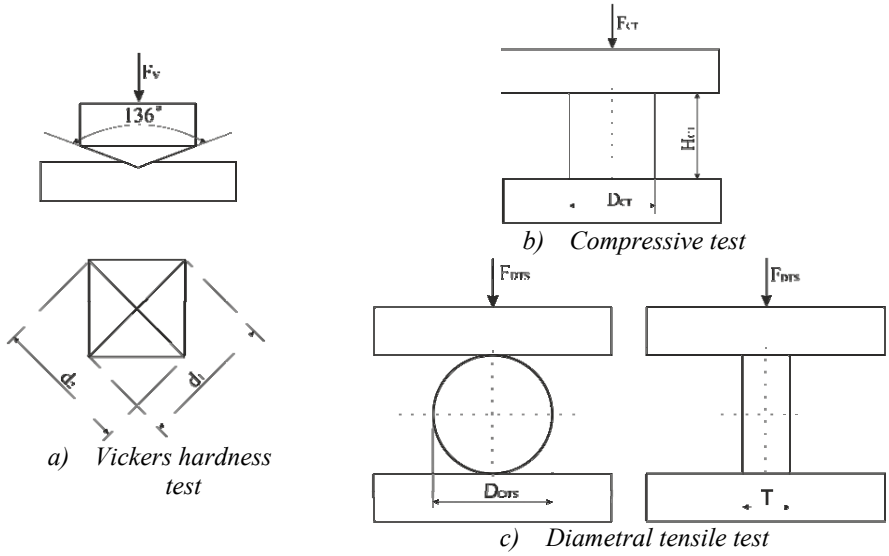
Bis-GMA- bisfenol A diglicidil ether dimethacrylate; Bis-EMA- bisfenol A polyethylene glycol dietherdimethacrylate; UDMA- urethane dimethacrylate; TEGMA- triethyleneglycoldimethacrylate; PEGDMA- polyethylene glycol dimethacrylate
*Dataobtainedfrom the manufacturers

Diametral tensile test is a common method for measuring tensile strength of brittle materials because it avoids some of the difficulties inherent in direct and flexural tensile testing [20]. For the diametral tensile test (*DTS)* three specimens of each material were used (figure 3c). The specimens were compressed diametrically introducing tensile stress in the material in the plane of the force application, figure 2c. No lubrication was applied. Press speed was 10mm/min. Compression was conducted by flat dies until the crack. Equation (3) was applied for diametral tensile strength *DTS*calculation.

$$DTS = \frac{2 \cdot F_{DTS}}{D_{DTS} \cdot T \cdot \pi} \left[\frac{N}{mm^2} \right] \tag{3}$$

where:

F_{DTS} – force occurred in experiment [N]
D_{DTS} =5mm - diameter of specimens [mm]
T=2mm - thickness of the specimens [mm]

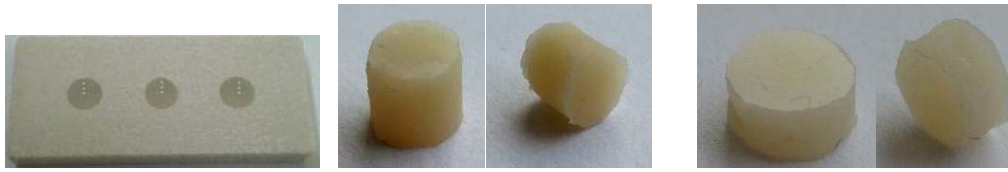


a) *Vickers hardness test*

b) *Compressive test*

c) *Diametral tensile test*

Fig. 2 *– Schemes of the processes*



a) *Vickers hardness test specimens*

b) *Compressive test specimens*

c) *Diamtral tensile test specimens*

Fig. 3 *– Specimens used in experiment*

3. RESULTS

Analysing the results, it can be concluded that the highest value of Vickers hardness had FZ550 and the lowest value occurred in the case of FUB(figure 5). Also it can be concluded that values of Vickers hardness do not differ significantly on top and bottom side of the specimens. That difference was approximately 3%.
In figures 6a and 6b, force – stroke diagrams obtained in compressivetest and diametral tensile test are presented.

a) *b)* *c)*

Fig. 4 *– Specimens after experiment*

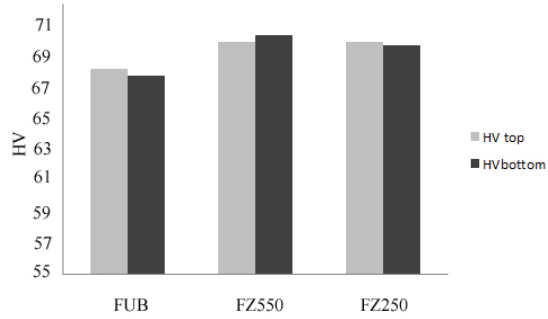
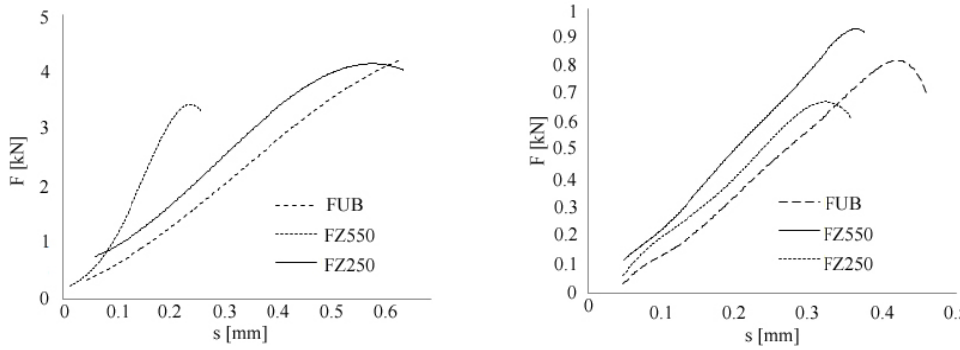


Fig. 5 *– Hardness test values*



a) Compressive test *b) Diametral tensile test*

Fig. 6 *– Forming stroke diagrams*

In the case of compressive test, the lowest value of loaded force was achieved in case of FZ550 (figure 6a).However, diametral tensile test showed that the highest force value (0.95kN) appeared in the case of FZ550 and the lowest value (0.65kN) in the case of FZ250 (figure 6b). Also it can be concluded that higher value of strokes were achieved in compressive test than in diametral tensile test. According to calculations the highest value of compressive strength *p* hadFZ250. However, the highest diametral tensile strength *DTS* had FZ550.

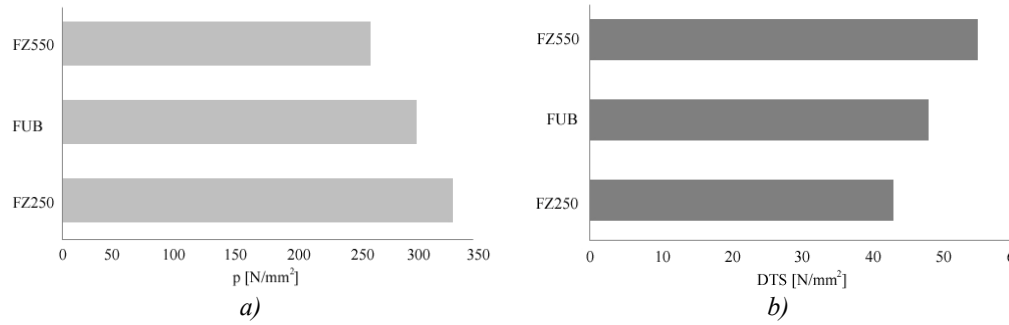


***Fig. 7** – Compressive strength (a) and diametral tensile test (b)*

4. CONCLUSION

Taking into account the forces that develop during chewing,all the measured values of materials strength in the study were satisfactory. This showed that all the tested materials can be used as universal materials in either frontal or lateral masticatory segments. However, ccompressive strength was higher for mycrohybrid (FZ250) than for nanocomposites (FUB and FZ550). That shows that microhybrid material can be still material of choice for the lateral occlusal region. On the other hand, diametral tensile strength was higher for nanocomposites, indicating nanocomposites for use in the frontal masticatory region, where the tensile forces are mostly presented. Vickers hardness values were very close and similar between all tested materials. Hardness results on the both sides of samples were not significantly different. The samples have been polymerized on the top and on the bottom, showing that recommended clinical procedure for using 2mm thick layers isacceptable for the tested contemporary materials.

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OTPORNOST STOMATOLOŠKIH NANOMATERIJALA NA STATIČKO OPTEREĆENJE

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REZIME

Poznavanje inženjersih svojstava stomatoloških materijala je od velikog značaja, jer upućuje na odgovarajući odabir materijala i na terapeutske odluke koje doprinose dugotrajnom kliničkom uspehu. Određivanje mehaničke otpornosti ovih materijala može pružiti informacije iz kojih se mogu izvesti smernice zakliničku praksu. Nanokompozitni materijali na bazi smola su najčešće korišćeni nanomaterijali u savremenoj stomatološkoj praksi. Očekuje se da su ovi materijali ojačani prisustvom nanočestica. Cilj prikazane studije je bio određivanje mehaničke otpornosti savremenih stomatoloških nanokompozitnih materijala za zubne ispune.
Ključnereči: *mehaničkeosobine, test aksijalnog sabijanaja valjka, test poprečnog sabijanja valjka-indirekni test zatezanja, test tvrdoćepoVikersu, stomatološki nanokompoziti.*