Tribological Journal BULTRIB Vol. 7, 2018
Papers from the 13th International Conference **BULTRIB '18**
25-27 October 2018, Sofia, Bulgaria
Society of Bulgarian Tribologists
FIT – Technical University of Sofia

EFFECTS OF DIFFERENT FINISHING PROCEDURE ONTO NANOMECHANICAL CHARACTERISTICS OF VENEERING CERAMIC

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Abstract: *This paper describes the nanomechanical characteristics of fluorapatite veneering ceramic (IPS e.max Ceram, Ivoclar Vivadent) treated with three different surface finishing procedures: polishing, glazing and grinding, using the Anton Paar nanoindentation. Also, AFM analysis of different surface finishing procedure was done in order to determine the 3D surface topography and roughness parameter Ra. The hardness (HV) and Young's modulus (E) of the surface structure were presented as results of nanoindentation measurements. Nanoindentation tests was done using Berkovich diamond pyramid and the experiment was organized in a 3x4 array. Indentation imprints were investigated using the optical and Atomic Force Microscopy. The obtained results show that the nanomechanical properties mostly depend of prepared surface finishing procedures.*

Key Words: *Nanoindentation, Fuorapatite veneering ceramic, different finishing procedure, AFM analysis.*

1. INTRODUCTION

The aesthetic and biomechanical properties of dental materials mostly depend of the distribution and particles size, as well as the concentration of the crystals in the structure of material itself [1]. Glass ceramics has become very important material in the last few years and one of the most used materials in dentistry, solely because of its excellent aesthetics, good mechanical properties and the longevity of the dental restoration itself [2].

IPS e.max Ceram is a veneering ceramic with excellent aesthetics results designed for use in conjunction with all-ceramic systems. IPS e.max Ceram is a homogeneous mixture of fluorapatite-containing glass ceramic and sintered glass powders. Veneers are thin «laminates» made of ceramic, which are attached to the tooth and which do not cover the entire tooth. Only a small amount of tooth structure is ground away for veneers. Veneering ceramics play a key role in the esthetics of a restoration. Their interplay of shade, translucency, and brightness allows for a lively appearance of the natural and restored dentition [1,3].

The mechanical properties of ceramic materials largely depend of the surface roughness and structural defects of the material itself. Porosity has a major influence on the mechanical properties of ceramic materials, where the mechanical properties of the material significantly decreasing with pronounced porosity [4]. The indentation test allows useful information's about mechanical properties of investigated material, such as hardness, Young's modulus, induced stresses, work hardening and residual thermal stresses [5,6].

The aim of this study is to identify the nanomechanical properties of veneering ceramic (*IPS e.max Ceram, Ivoclar Vivadent*), under different finishing procedure (polishing, glazing and grinding), using the Anton Paar Nanoindentation. The obtained results of nanoindentation measurements were performed in order to define the hardness (HV) and the Young's modulus (E) of the surface structure as a function of the applied indentation load. Also, AFM analysis was done in order to determine the 3D surface topography and roughness parameter Ra, for all prepared samples.

2. EXPERIMENTAL PROCEDURE

2.1. Material and samples preparation

Veneering ceramic *IPS e.max Ceram* consists of a multicomponent system SiO_2-Li_2O-Na_2O-K_2O-ZnO-Al_2O_3. The glass structure of fluorapatite is additionally strengthened by a certain wt. of the components CaO, P_2O_5 and F, (Figure 1a). These three basic components are a prerequisite for the formation of a fluorapatite crystal $Ca_5(PO_4)_3F$. Thus, the formed fluorapatite content has a big influence on the natural aesthetic appearance of restoration in the form of reflection, trasuency and opalescence. The main component of the structure is SiO_2 with ~ 60% of wt. Chemical composition of commercial Veneering ceramic *IPS e.max Ceram* is given in Table 1 [2,7].

a) b)

Fig. 1. *IPS e.max Ceram veneering ceramic.*

Table 1*. Chemical composition of Veneering ceramic IPS e.max Ceram [3].*

Standard composition	(in % by weight)
SiO_2	60 – 65
Al_2O_3	8 – 12
Na_2O	6 – 9
K_2O	6 – 8
CaO	1 – 3
ZnO	2 – 3
Li_2O	1 – 2
ZrO_2	1 – 1.5
F	1 – 2
+ Others oxides	0.5 – 7

The preparation of samples required the mold manufacturing (diameter 20 mm and height 5 mm). Before testing, all samples were firstly sintered at prescribed temperature according to manufacturer's instructions (*Ivoclar Vivadent*). After sintering, the contact surfaces of samples are prepared with 3 different finishing procedures (polishing, glazing and grinding), Figure 1b.

The 1st sample was polished on the polishing machine, under controlled speed, using diamond sandpaper with different grits (280, 400, 600, 800, 1200 and 2000) with hand pressure and water cooling. The fine polishing was done after that by using the liquid emulsion with grain size of 6 and 0.04 µm. Final roughness prior to testing was Ra=12.239 nm. The contact surface of the 2nd sample was glazed according to the manufacturer's recommendations (*Ivoclar Vivadent*). Testing roughness was Ra=17.253 nm. On the end, contact surface of the 3rd samples was grinded by using diamond borer (Medin, ISO: 806 314 146 534 016, 150 µm – max). Measured roughness of grinding sample was Ra=0.786 µm.

On the end, all samples surfaces were cleaned with 70 % alcohol before every test, in order to remove any remaining surface contaminants.

2.2. AFM analysis

All results of roughness parameter (Ra) and 3D surface topography were obtained by NT-MDT AFM microscope (Fig. 1). The measurement range on all samples is 100x100 µm. AFM have been described thoroughly in a previous publication [8].

2.2 Nanoindetation

Nanoindentation tests were done using Anton Paar Nanoindenter, which is located at the Tribology center on the Faculty of Engineering in Kragujevac (Figure 2).

Fig. 2. *Anton Paar Nanoindenter [1].*

Indentation data were obtained using loads of 50, 100, 200 and 400 mN for each samples. Maximum load holding time was 10 s. A Berkovich diamond indenter was used for all indentations. Each test was repeated three times. The study obtained values of hardness and Young's modulus.

Indentation imprints were investigated using the Optical and AFM microscopy.

3. RESULTS AND DISCUSION

Nanomechanical tests were preceded by the AFM analysis in order to determine the roughness parameters Ra and 3D topography of the tested material under different surface finishing procedure (Figure 3). Presented results, show that lowest values of R_a have polished finishing procedure, as expected.

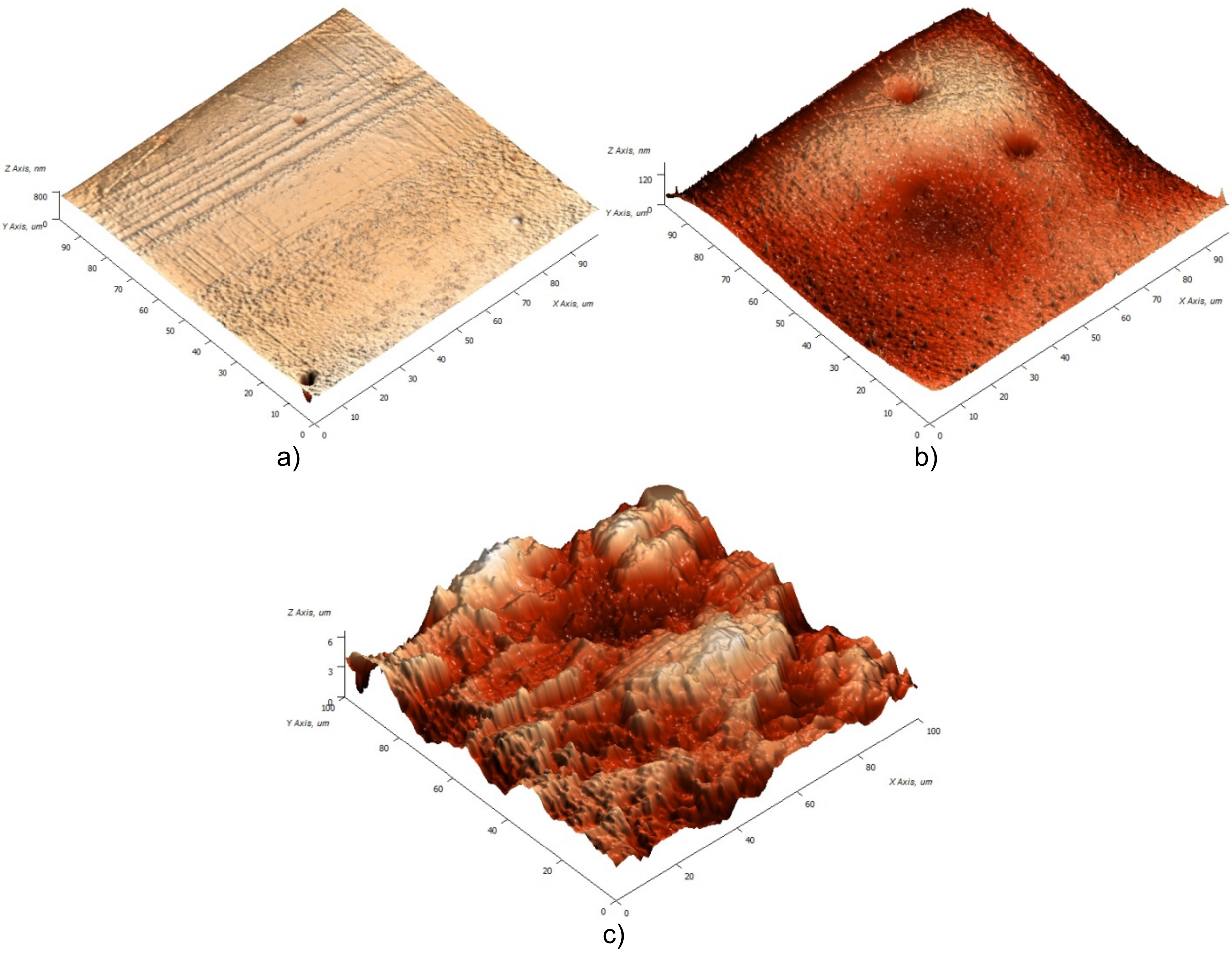
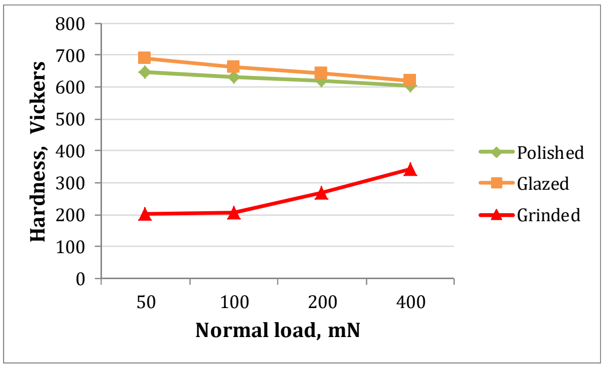


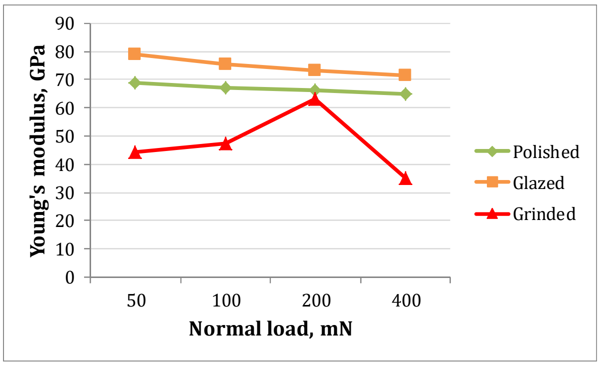
Fig. 3. *AFM analysis (3D topography) of samples: a) polished, b) glazed, and c) grinded surface.*

Surface roughness has a big influence on the many things as well as aesthetic of the contact surface of material itself, changing color on the dental restoration, secondary caries and gingival irritation, and the mutual wear of contact surfaces of teeth and antagonists (natural tooth or dental restoration). The most main goal in esthetic dentistry is that the finishing procedure of the contact surface of material should be as smooth as possible [1,2,8].

The obtained results of nanoidentation (hardness and Young's modulus) are presented at the Fig. 4. Results are presented as mean values of all measured parameters obtained as the arithmetic mean of the 3 repeated tests.



a)

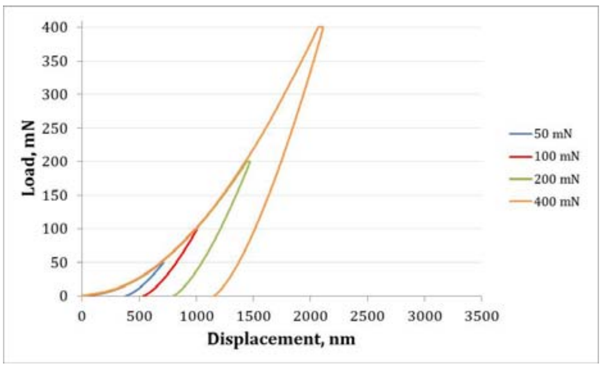


b)

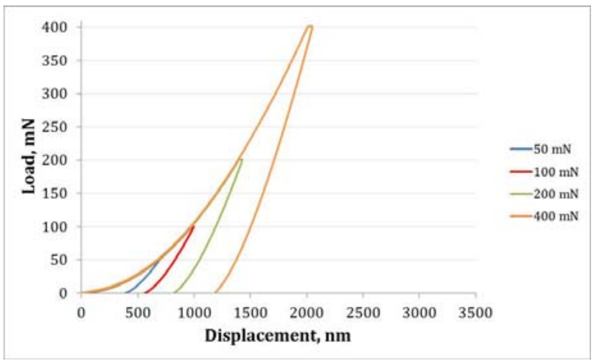
Fig. 4. *Nanoindentation results: a) Hardness (HV) and (b) Young's modulus (E).*

From the presented Figure 4 it can be clearly seen that the highest value of hardness and Young's modulus has a glazed sample. Also, is visible trend for polished and glazed surfaces that hardness and elastic modulus decreases in a very small range with increasing normal load. Grinding does not have a trend as polished and glazed surfacesses probably due to the pronounced surface roughness at the place of testing. The contact between the Berkovich indenter and the surface of the sample is realized mostly per tops of roughness. The phenomenon of decreasing hardness by increasing the indentation load is known under the term "Indentation size effect (ISE)" [10,11]. Figures 4a and 4b clearly show that the glaze has a significant impact on the obtained results, ie, provides better mechanical properties of the material itself.

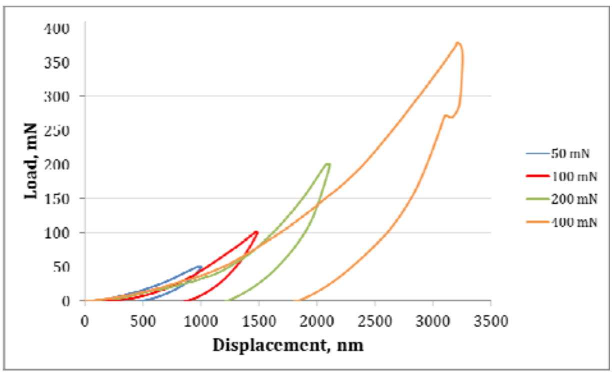
Figure 5 shows the load-displacement curves for different prepared samples as mean values of three indentations for loads 50, 100, 200 and 400 mN. The curves have proper form and clearly show that it is maximum load holding time properly selected [12,13].



a)



b)

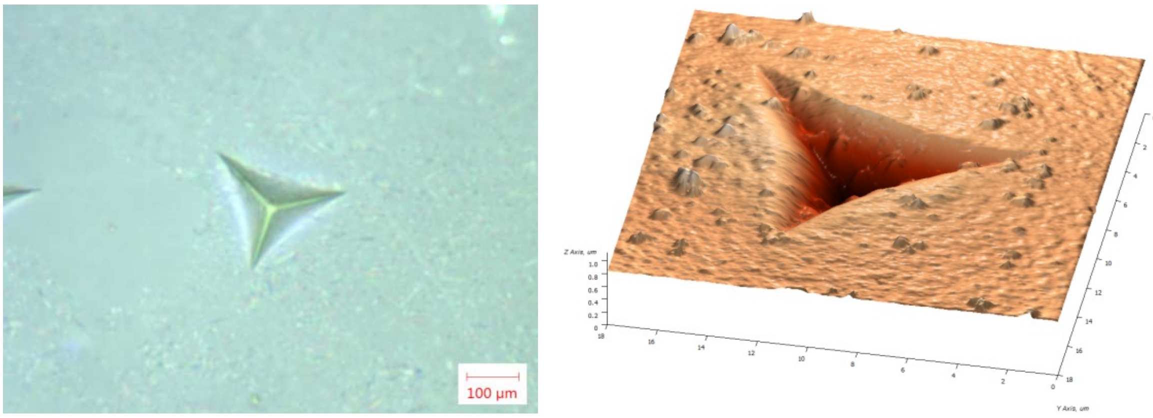


c)

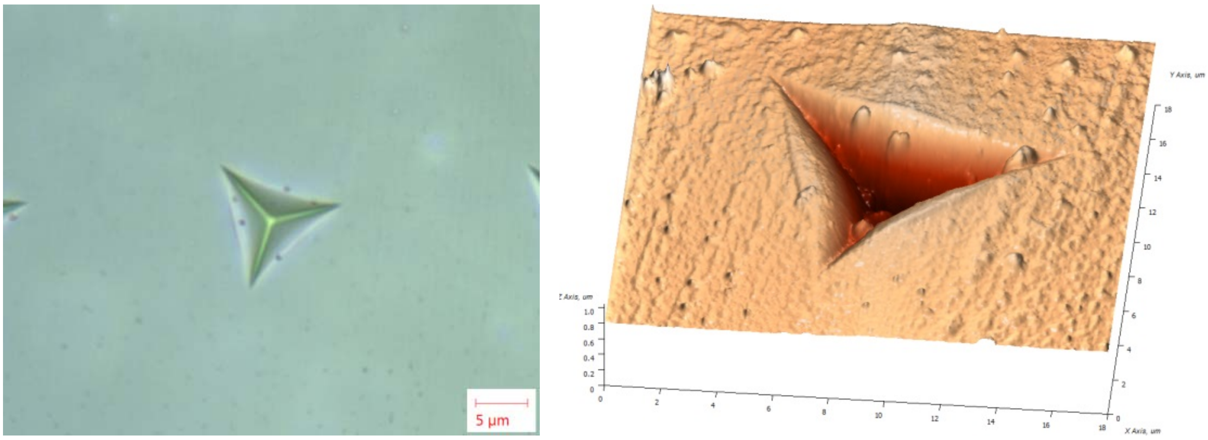
Fig. 5. *Load-displacement curves under different finishing procedure: a) polished, b) glazed and c) grinded.*

The diagrams (Figure 5) clearly show that the indentation depth proportionally increasing with the increase of normal load. There are no major differences in indentation curves for polished and glazed tested samples, while the grinding curve has a mild deviation from the previous two samples, especially in the case of a 400 mN indentation load, where is a irregular shape of the load-displacement curve result of different surface roughness in the contact zone and gas bubble occurrence in contact suraface of samples, whose presence is characteristic for this material [1].

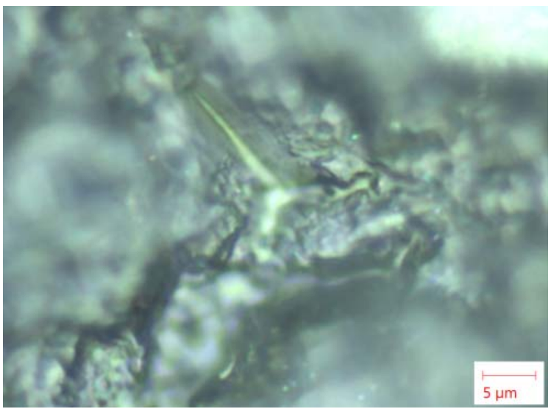
The Figure 6 shows representative indentation imprints (400 mN) of Veneering ceramic under different finishing procedure, obtained on optical (x100) and Atomic force microscopy. Nanoindentation on grinding sample was presented just by optical microscopy because it was impossible to find indentation imprints on AFM due to their small size of imprints and big surface roughness of the material.



a)



b)



c)

Fig. 6. *Indentation imprints at load of 400 mN, analysed by optical (left, x100) and AFM microscopy (right): a) polished; b) glazed and c) grinded surface.*

Indentation imprints are clearly formed with visible edges in the surface layer of material. On Figures 6a and 6b around imprints it can be noticed mild plastic deformation (brighter zone), as a result of displacement of material (piling-ups) during the penetration of indenter.

Plastic creep of material on the along the side of the indentation marks can be considered as the basic physical process softening the material due to the phenomenon of shear [14]. The reason for this is that the plastic creep of material by shearing causes certain structural changes in the field of the material itself, which means that the deformation in that zone is much faster than in the other zone of the material [15]. Materials that move from the piling-up condition to the sinking-in condition, become much more elastic [16]. This also shows the importance of the Young's modulus, which present a measure of the stiffness of the material. Since the polished and glazed surfaces are most common in practice, mechanical properties of materials has big importance on lifetime of dental restoration because they mostly depends on quality of the finishing procedure.

Presented results are in accordance with the obtained results of several similar studies [15,17-21], also they may assist in better understanding of the mechanical behaviour of Veneering ceramic under different finishing procedure and thus facilitate the design and CAD/CAM manufacture for dental restorations.

5. CONCLUSION

The mechanical properties of ceramic materials largely depend of the surface roughness and structural defects of the material itself. Based on the obtained results, it can be concluded that nanomechanical properties mostly dependent of applied surface finishing procedures.

The highest value of hardness and Young's modulus has a glazed sample. The glaze, as a finished procedure, has a significant impact on the obtained results, ie, provides better mechanical properties of the material itself.

Presented results may assist in better understanding of the nanomechanical behaviour of veneering ceramic under different finishing procedure and thus facilitate the design, selection and CAD/CAM manufacture for dental restorations.

ACKNOWLEDGMENTS

Research presented in this paper was supported by the Ministry of Education, Science and Technology Development of Republic of Serbia, Grant TR-35021.

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