Serbian Tribology Society

SERBIATRIB '19

16th International Conference on Tribology

Faculty of Engineering University of Kragujevac

Kragujevac, Serbia, 15 – 17 May 2019

NANOINDENTATION OF ZIRCONIUM DENTAL CERAMIC PREPARED WITH DIFFERENT FINISHING TECHNIQUES

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Abstract: *This paper describes the nanomechanical characteristics of Zirconium (IPS e.max ZirCAD, Ivoclar Vivadent) treated with three different surface finishing techniques: polishing, glazing and grinding, using the Anton Paar nanoindentation. The hardness (HV) and Elastic modulus (E) of the surface structure were presented as results of nanoindentation measurements. Nanoindentation tests were 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. Also, AFM analysis was used in order to present the 3D surface topography and roughness parameter Ra. The obtained results show that the nanomechanical properties mostly depend of finishing technique.*

Keywords: *Zirconium, Nanoindentation, different finishing techniques: polishing, glazing, grinding.*

1. INTRODUCTION

The history of dental ceramics dates back to the period of ancient pharaohs, when dental crowns and prosthesis were made of glass or ivory and with the help of gold wire fixed at their places [1].

The increased demand for aesthetics led to the development of all ceramic restorations. The first use of zirconia in dentistry was present in the production of dental pins, fixed prosthesis and dental implants, and very often in the literature it was possible to find the term "white steel" [2,3]. However, the real breakthrough in aesthetic dentistry had come with the development of CAD/CAM technology [4]. Zirconium in combination with new CAD/CAM technology is increasingly being used and developed, due to its excellent characteristics of material, as well as due to the precision and rapid production. The production of dental restorations in this way allows perfect adherence of crowns and bridges to the gingiva, thus providing patients with excellent quality, comfort and longevity of compensation in all load zones [5,6].

The surface roughness and structural defects have big impact on mechanical properties of ceramic materials [1]. It is known that more favourable conditions are made with high roughness of 0.2 mm for higher appearance of tooth plaque and cavities [7]. On the other side, the smooth surface of the intraoral structure ensures the comfort of the patient and facilitates oral hygiene [8-10]. Moreover, inadequate polishing of the contact surface of the restoration can lead to residual surface roughness, which directly disturbs the mechanical and aesthetic characteristics of the contact surface of the material itself [11-13].

The aim of this study is to identify the nanomechanical properties of Zirconium (IPS e.max ZirCAD, Ivoclar Vivadent), under different finishing techniques (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 Elastic modulus (*E*) of the surface structure as a function of the applied indentation load.

2. EXPERIMENTAL PROCEDURE

2.1 Material and samples preparation

IPS e.max ZirCAD is a presintered yttriumstabilized zirconium oxide block (Y - TZP) for the CAD/CAM technology (Fig. 1a). There are different sizes of *IPS e.max ZirCAD* blocks depending on the oral zone and the type of restoration that is being performed. The block is white, chalk structure and in poisoned state it is distinguished by porous morphology (50%). The hardness of the raw material is very small, which enables fast and easy processing of the block on the CAD/CAM system in the desired shape. After forming, the material is sintered in a high temperature furnace specially developed for oxide ceramics at a temperature of 1500 ° C. During the 8-hour sintering process, the crystals form the final tetragonal homogeneous structure and obtain their final bending strength of over 900 MPa. Chemical composition of *IPS e.max ZirCAD* is given in Table 1 [14].

Table 1. Chemical composition of *IPS e.max ZirCAD* [15]

Standard composition	(in % by weight)
ZrO2	87 – 95
Y2O3	4 – 6
HfO2	1 – 5
Al2O3	0.1 – 1

Experimental investigations were realised on 3 samples with dimensions 18x14x12 mm. After sintering, the contact surfaces of samples are prepared with 3 different finishing techniques (polishing, glazing and grinding), Figure 1b. Finishing techniques have been described thoroughly in a previous publication [16].

a)

b)

Figure 1. a) *IPS e.max ZirCAD* ceramic; b) samples prepared with 3 different finishing techniques

The surface of the samples was cleaned with 70 % alcohol using a soft cotton cloth, in order to remove any remaining surface contaminants. After that, samples were ultrasonically cleaned in distilled water for 30 min, and allowed to dry at room temperature.

2.1 Surface roughness

Nanomechanical tests were preceded by the AFM analysis in order to determine the roughness parameters Ra and 3D topography of each sample (Figure 2). Surface roughness was measured by AFM of NT-MDT manufacturers, which is located at the Tribology center on the Faculty of Engineering in Kragujevac. The measurement range on all samples is 100x100 µm and the surfaces roughness is measured along the same reference length. The obtained results of roughness parameter Ra is presented in Table 2.

Table 2. Comparative view of R_a under different finishing techniques of Zirconium

Measuring range, 100x100 µm	Roughness parameter, R_a
The polished surface	10.728 nm
The glazed surface	16.655 nm
The grinded surface	0.415 µm

Presented results, show that lowest values of Ra have polished finishing technique, as expected. It should be noted that aesthetic dentistry has always strived for material contact surface to be as smooth as possible [1].

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.

Table 3 shows the defined conditions of nanoindentation test.

Table 3. Defined conditions of nanoindentation test

Test method
- Berkovich three-sided diamond pyramid - 3x4 array - Ambient temperature: 23±2 °C
Loads
- 50, 100, 200 and 400 mN
The loading and unloading rate
- 100 mN/min for load of 50 mN - 200 mN/min for load of 100 mN - 400 mN/min for load of 200 mN - 800 mN/min for load of 400 mN
Maximum load holding time
- 10 s

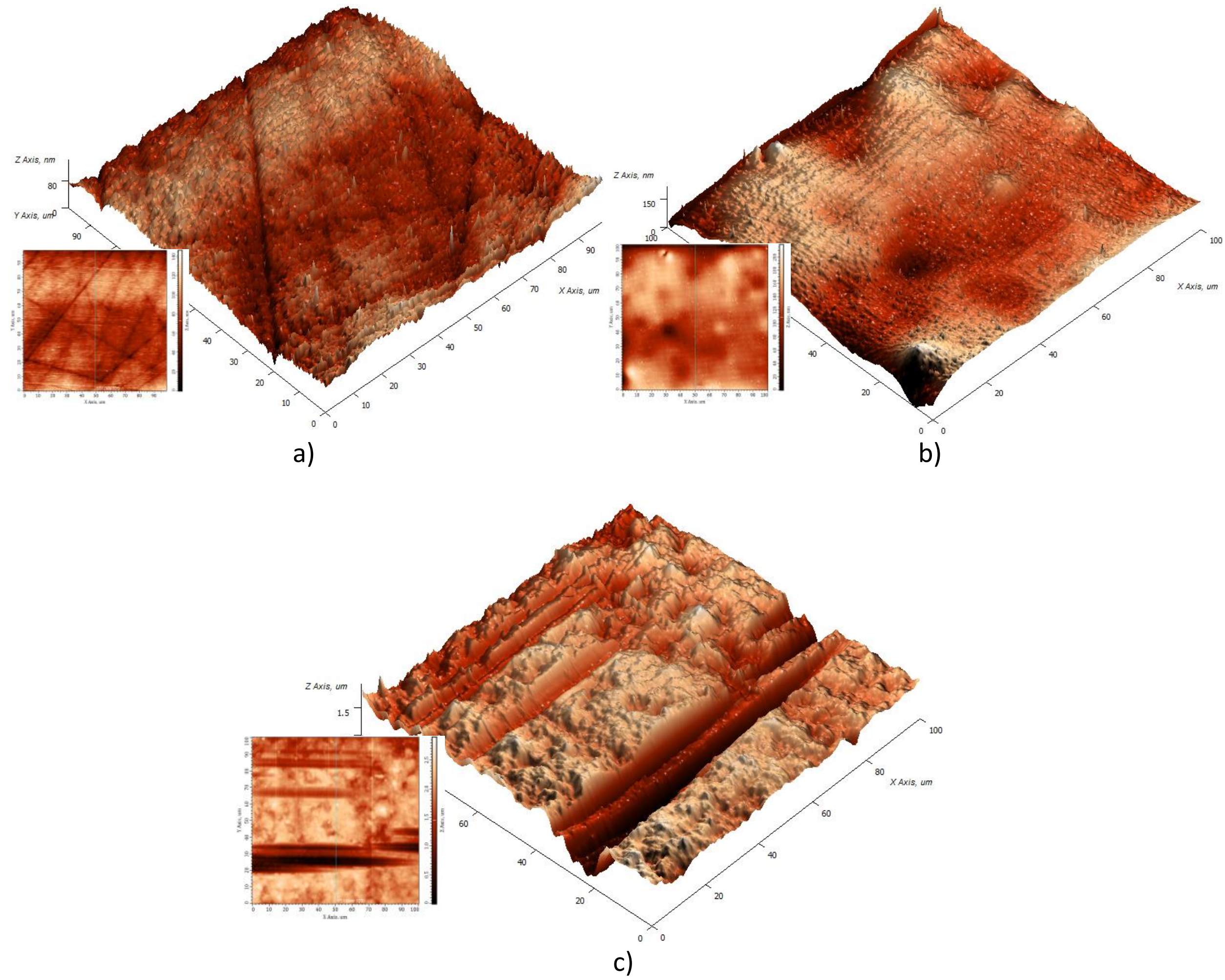
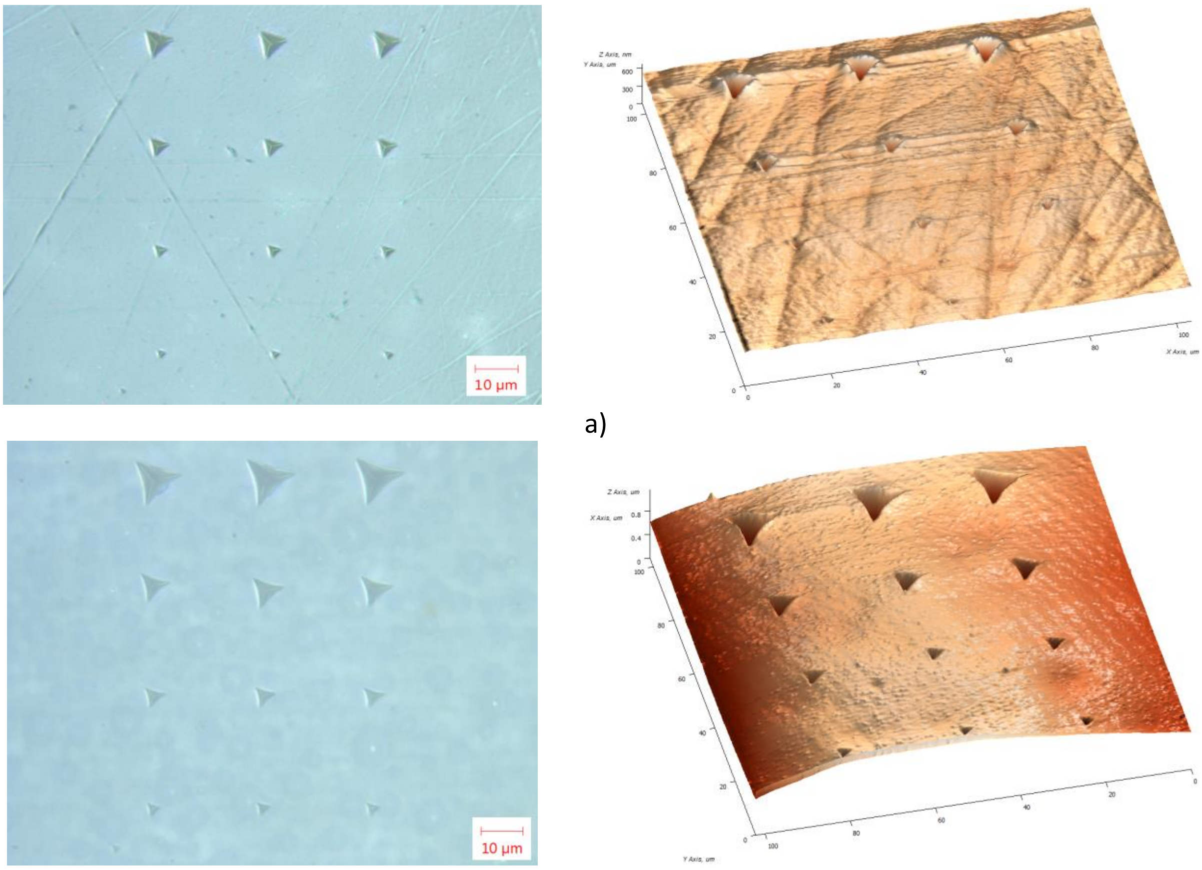


Figure 2. AFM analysis (3D topography) of samples: a) polished, b) glazed, and c) grinded surface



a)

b)

Figure 3. Indentation view of 3x4 array: a) polished and b) glazed surface

Each test was repeated three times, because the experiment was organized in a 3x4 array (Fig. 3). Distance between centres of imprints was 30 μm, it was taken into account that imprints were not too close to each other to avoid influence of work hardening on mechanical properties of tested material. The Poisson's ratio for LGC was 0.300 [1].

A Berkovich diamond indenter (three-sided pyramidal) was used for all indentations. Indentation imprints were investigated using the Optical and AFM microscopy.

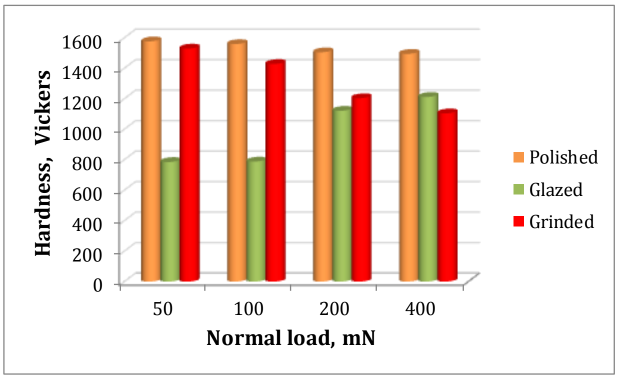
3. RESULTS AND DISCUSION

The obtained results of nanoidentation values, hardness (*HV*) expressed in Vickers units and elastic modulus (*E*) are presented in Table 4.

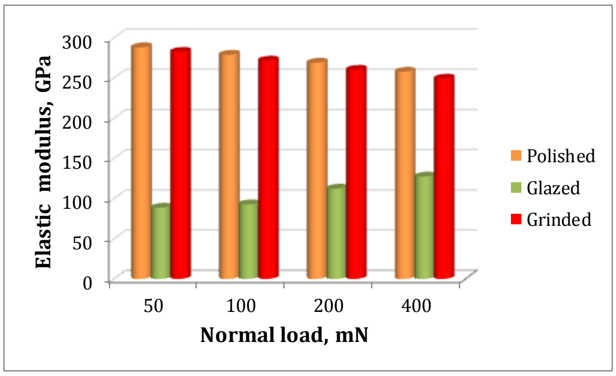
In order to better understand the shown values in Table 4, the Figure 4 presents a histogram comparison of the obtained Hardness and Elastic modulus results of the tested material. From the Figure 4 it can be clearly seen that the highest value of hardness and Elastic modulus has a polished surface. Also, is visible trend for polished and grinded surfaces that hardness and elastic modulus decreases in a small range with increasing indentation load.

Table 4. Mean values of indentation process performed on Zirconium (*IPS e.max ZirCAD*) prepared with different finishing techniques

IPS e.max ZirCAD	Loads of indentation							
	50 mN	100 mN	200 mN	400 mN	50 mN	100 mN	200 mN	400 mN
	Hardness *(HV)*, Vikers				**Elastic modulus *(E)***, [GPa]			
The polished surface	1576.17	1558.33	1504.53	1494.10	288.17	278.85	269.03	257.72
The glazed surface	784.65	787.36	1120.46	1211.60	88.51	92.80	112.32	127.35
The grinded surface	1528.33	1427.40	1203.43	1103.84	282.51	271.64	260.16	249.12



a)

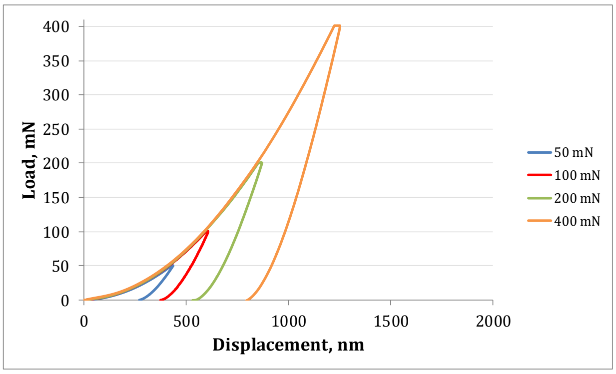


b)

Figure 4. Nanoindentation results: a) Hardness (*HV*); and (b) Elastic modulus (*E*)

The phenomenon of decreasing hardness by increasing the indentation load is known under the term "Indentation size effect (ISE)" [17,18].

Figures 4a and 4b clearly show that the glaze has a significant impact on the obtained results, i.e., provides poorer mechanical properties of the material itself. Glazed sample does not have a trend as polished and grinded surfaces, the trend of decreasing the hardness and elastic modulus with the increase of the indentation load is completely diametric compared to the results of the polished and grinded surfaces. In the case with smaller indentation loads (50 and 100 mN), the hardness value is well below the real hardness of the material itself (~785 Vickers). The thickness of the glaze has a large influence on the obtained results, because the penetration depth of the indenter into the glaze surface is much bigger than in the base material. The measured values of the indenter penetration depth, ranging from 670-950 nm with the indentation loads of 50 and 100 mN, are insufficiently large to properly characterize the real value of the hardness of the base material. The same conclusion can be attributed to the results of the Elastic modulus for the glazed surface, because the obtained results are characterized by the same trend as the results of the measured hardness values. Based on that it can be concluded that the obtained values of hardness and elastic modulus of glazed zirconium can be neglected when applying selected values of indentation loads.



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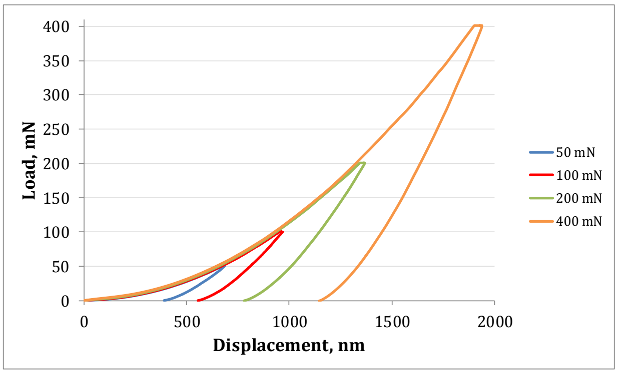

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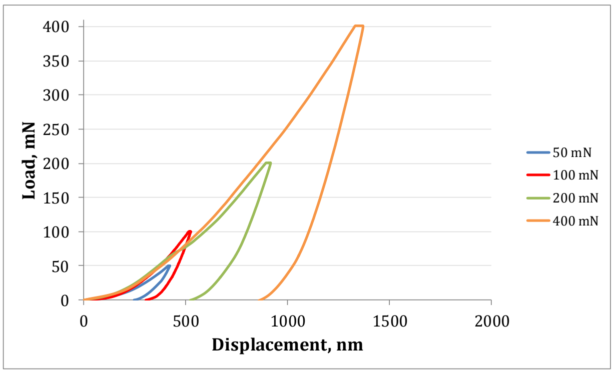


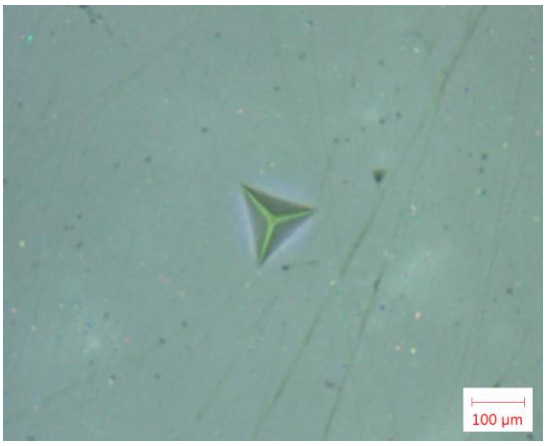
c)

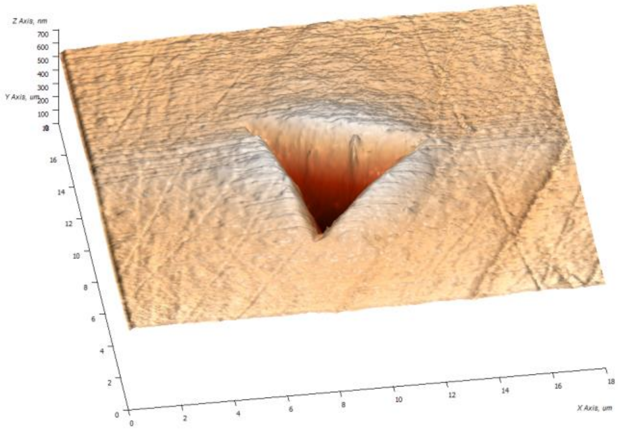
Figure 5. Load-displacement curves under different finishing techniques: a) polished; b) glazed; and c) grinded

The Figure 5 shows the load-displacement curves for different prepared samples as mean values of three indentations for loads of 50, 100, 200 and 400 mN. The curves have proper form and clearly show that it is the maximum load holding time properly selected [19,20].

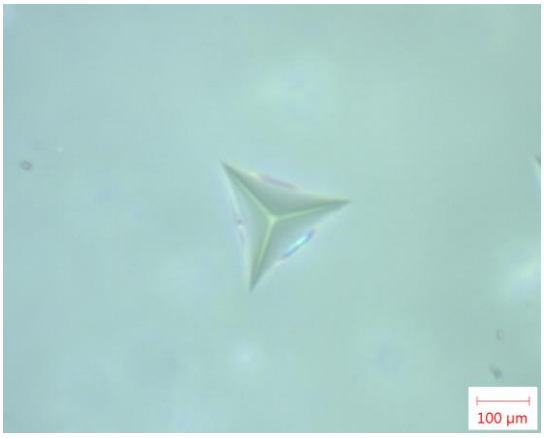
The diagrams (Figure 5) clearly show that the indentation depth proportionally increases with the increase of indentation load. There are no major differences in indentation curves for polished and grinded tested samples, while the glazed curve has a mild deviation from the previous two samples. The glazed finishing treatment has a significantly higher value of the load-displacement (maximum indentation depth) compared to other finishing techniques [1].

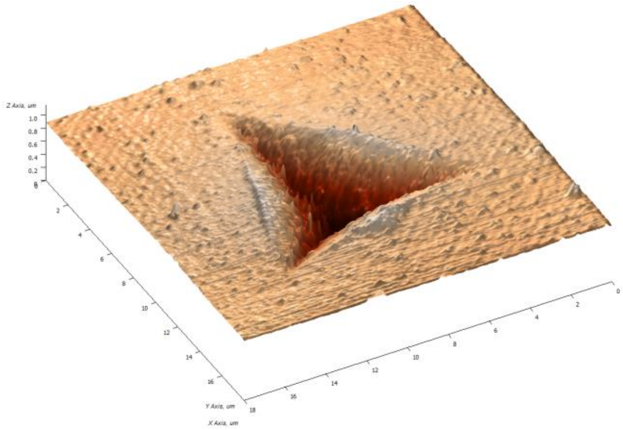
The Figure 6 shows representative indentation imprints (400 mN) of Zirconium under different finishing techniques, obtained on optical (x100) and AFM microscopy. Nanoindentation on grinding sample was presented just by optical microscopy because it was impossible to find indentation imprints on AFM due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





a)





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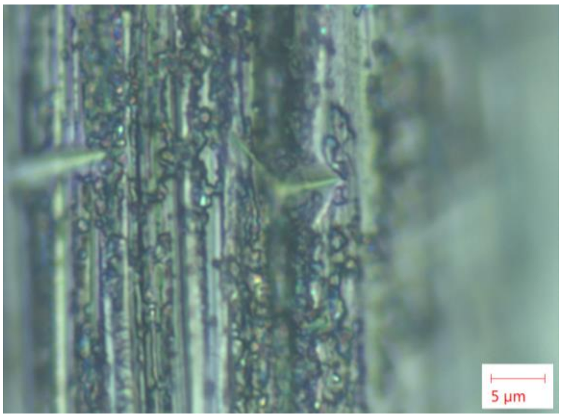

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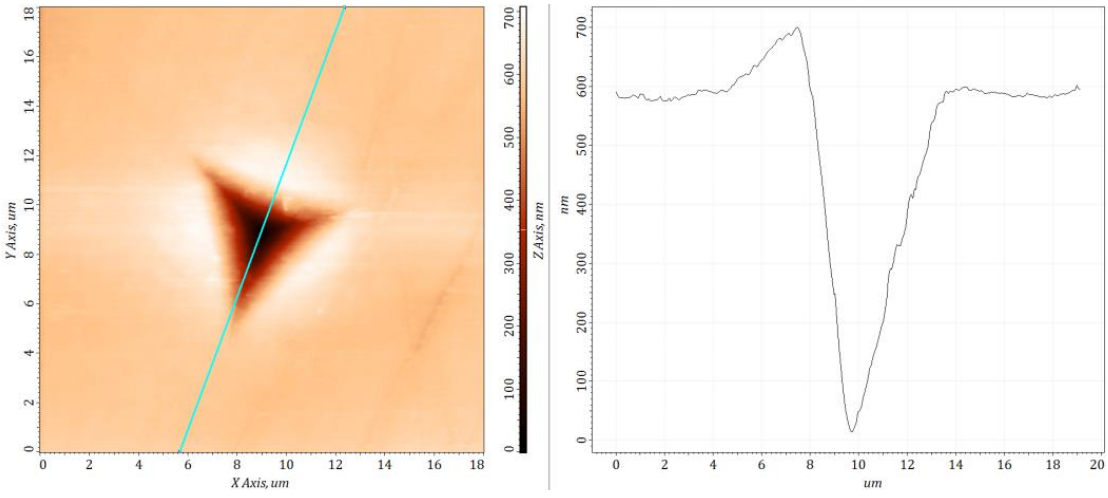
Figure 6. Indentation imprints at load of 400 mN, analysed by optical (left, x100) and AFM microscopy (right): a) polished; b) glazed and c) grinded surface



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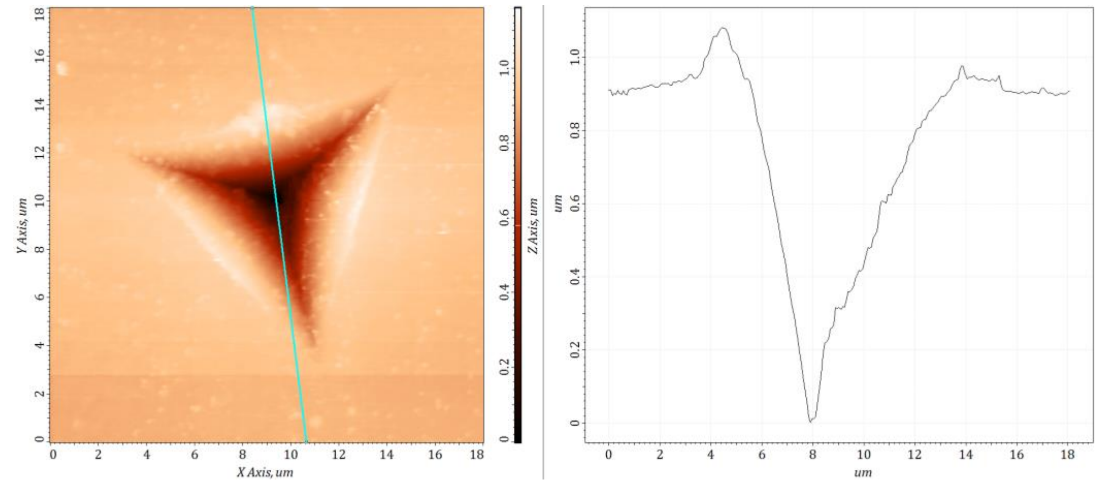

b)

Figure 7. 2D view AFM analysis of nanoindentation and cross-section of the imprints depth profile, under indentation load of 400 mN, a) polished, and b) glazed surface

Indentation imprints are clearly formed with visible edges in the surface layer of material. A mild plastic deformation around imprints can be seen in Figures 7a and 7b (brighter zone), as a result of displacement of material (piling-ups) during the indenter penetration.

Material plastic creep along the side of the indentation marks can be considered as the basic physical process which softens the material due to the phenomenon of shear [21]. This is why the material plastic creep, 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 [22]. Materials that move from the piling-up condition to the sinking-in condition become much more elastic [23]. This also shows the importance of the Elastic modulus, which present a measure of the material stiffness. Since the polished and glazed surfaces are most common in practice, therefore a mechanical property of materials has big importance on lifespan of dental restoration because they mostly depend on quality of the finishing techniques.

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 depend on applied surface finishing techniques.

The polished sample has the highest value of Hardness and Elastic modulus. The obtained results of hardness and elastic modulus of glazed zirconium can be neglected when applying selected values of indentation loads (50, 100, 200 and 400 mN), because of the unrealistic obtained values. The thickness of the glaze has a large influence on the obtained results, because the penetration depth of the indenter into the glaze surface is much bigger than in the base material.

The presented results may be helpful in better comprehension of the nanomechanical behaviour of dental ceramic based on zirconium under different finishing techniques and thus facilitate the design, selection and CAD/CAM manufacture for dental restorations.

ACKNOWLEDGMENTS

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

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