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IN VIVO STUDY OF THE NANOMECHANICAL PROPERTIES OF LEUCITE GLASS CERAMIC PREPARED WITH DIFFERENT SURFACE FINISHING PROCEDURES

Abstract: *This paper reports the nanomechanical properties of Leucite glass ceramic prepared with three different surface finishing procedures: polishing, glazing and grinding, using the Nanoindenter. Also, AFM analysis was done in order to determine the roughness parameter Ra. The nanoindentation were performed in order to define the hardness (HV) and Young's modulus (E) of the surface structure 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 of nanomechanical properties mostly depend of applied surface finishing procedures.*
Key words: *Leucite glass ceramic (LGC), different finishing procedure, nanoindentation, 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]. Leucite glass ceramic (LGC) is all-ceramic system which is characterized by high aesthetic quality thanks to the high and flexible translucency of the material itself. LGC have flexural strengths of 120-150 MPa and material usually can be used in the dental restoration such as veneers, inlays, onlays and one anterior crowns [2,3].

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 Leucite (*IPS e.max CAD, Ivoclar Vivadent*), prepared with three different finishing procedures: polishing, glazing and grinding, using the *Anton Paar* Nanoindenter. 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.

2. EXPERIMENTAL PROCEDURE

2.1 Material and sample preparation

Commercial material IPS Empress CAD (*Ivoclar Vivadent, Lichtenstein*) is all-ceramic material based on the leucite phase system SiO_2-Al_2O_3-K_2O designed for CAD/CAM technology. Chemical composition of Leucite is: SiO_2 60-65%wt., Al_2O_3 16-20 %wt., K_2O 10-14 %wt., Na_2O 3.5-6.5 %wt., others oxides 0.5-7 %wt. and pigments 0.2-1 %wt.

Experimental investigations were realised on 3 samples with dimensions 18x14x12 mm.

Before testing, all samples were firstly crystallized in a furnace at a prescribed temperature according to manufacturer's instructions. The contact surfaces of samples were prepared with different finishing procedure: polishing, glazing and grinding. The first sample was polished using different diamond sandpaper (280, 400, 600, 800, 1200 and 2000) by polishing machine. After that was done fine polishing using the liquid emulsion with grain size of 6 and 0.04 μm (DP-suspension, and the O-M In Suspension). The contact surface of the second sample was glazed according to the manufacturer's recommendations (with vacuum, 400 °C entry temperature, 730 °C high temperature for 1 min) [7]. On the end, contact surface of the third samples was grinded by using diamond borer (Medin, ISO: 806 314 146 534 016, 150 μm – max).

The surface of the all samples was cleaned with 70 % alcohol before every test, in order to remove any remaining surface contaminants.

2.2 Nanoindetation

Nanoindentation of LGC samples was done using *Anton Paar* Nanoindenter.

Table 1 shows the defined conditions of nanoindentation test. The experiment was realized in a 3x4 array and hence each test was repeated three times. Distance between centres of imprints was 30 μm. The Poisson's ratio for LGC is 0.225 [8].

Table 1. Defined conditions of nanoindentation test

Test method
- Berkovich three-sided diamond pyramid - 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

The study obtained values of Hardness (HV) and Young's modulus (E) on all samples. All indentation imprints were analysed by 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 of polished surface (Ra = 6.773 nm), glazed surface (Ra = 19.708 nm) and grinding surface (Ra = 0.686 µm). AFM have been described thoroughly in a previous publication [9].

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 [2,8].

The Figure 1 shows the obtained results of nanoidentation values, hardness (HV) and Young's modulus (E). Results are presented as mean values of all measured parameters obtained as the arithmetic mean of the three repeated tests.

From the presented Figure 1 it can be clearly seen that the highest value of hardness and Young's modulus has a glazed sample. Also, Figure 1 shows clearly 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.

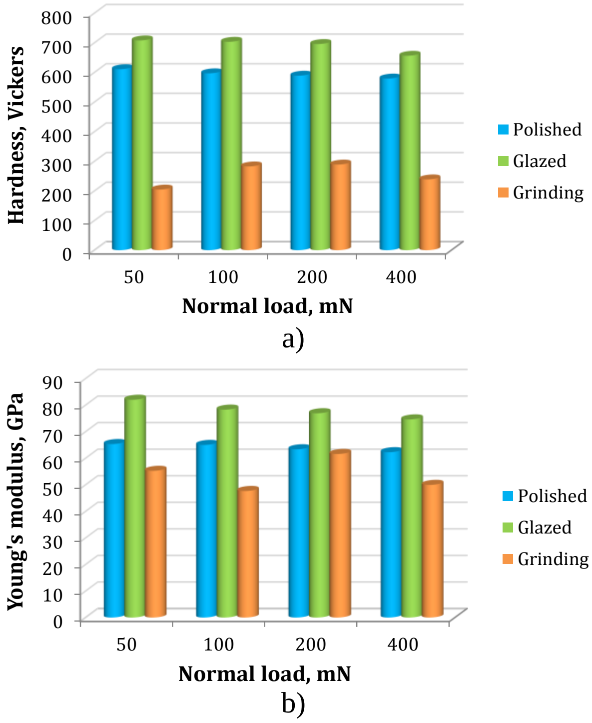

Fig. 1. Nanoindentation values of LGC under different finishing procedures: a) Hardness (HV) and (b) Young's modulus (E).

The phenomenon of decreasing hardness by increasing the indentation load is known under the term "Indentation size effect (ISE)" [10,11]. Figures 1a and 1b clearly show that the glaze has a significant impact on the obtained results, ie, provides better mechanical properties of the material itself. The mean value of glazed hardness is ~ 70 Vickers larger compared to the polished surface.

Figure 2 shows the load-displacement curves for different prepared samples of LGC 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].

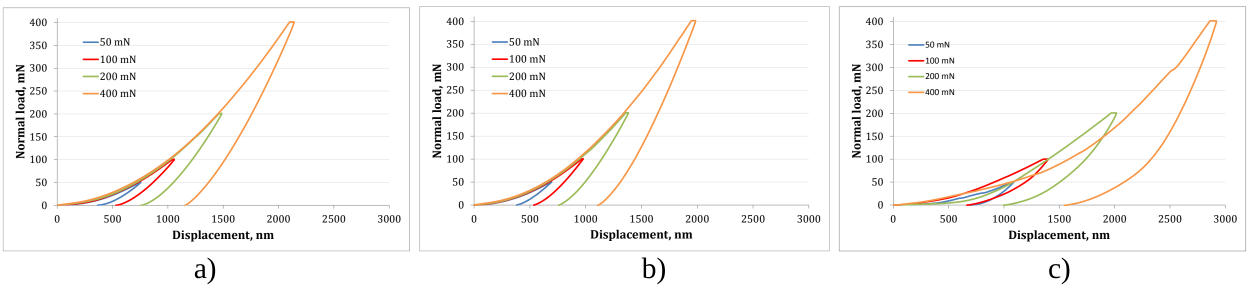

Fig. 2. Load-displacement curves for LGC under: a) polished, b) glazed and c) grinding.

The Figure 2 show that the indenter displacement is proportionally increasing with the increase of normal load. It can be clearly seen form presented curves that there is no differences in indentation curves for polished and glazed tested samples (Figures 2a and 2b), while the grinding curve (Figure 2c) has a mild deviation from the previous two surfacesses. What is obvious is that the different surface roughness in the contact zone have a big impact on the obtained results.

Figure 3 shows representative indentation imprints (400 mN) of LGC 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.

Indentation imprints are clearly formed with visible edges in the surface layer of material. On Figures 3a and 3b 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.

a)

b)

c)

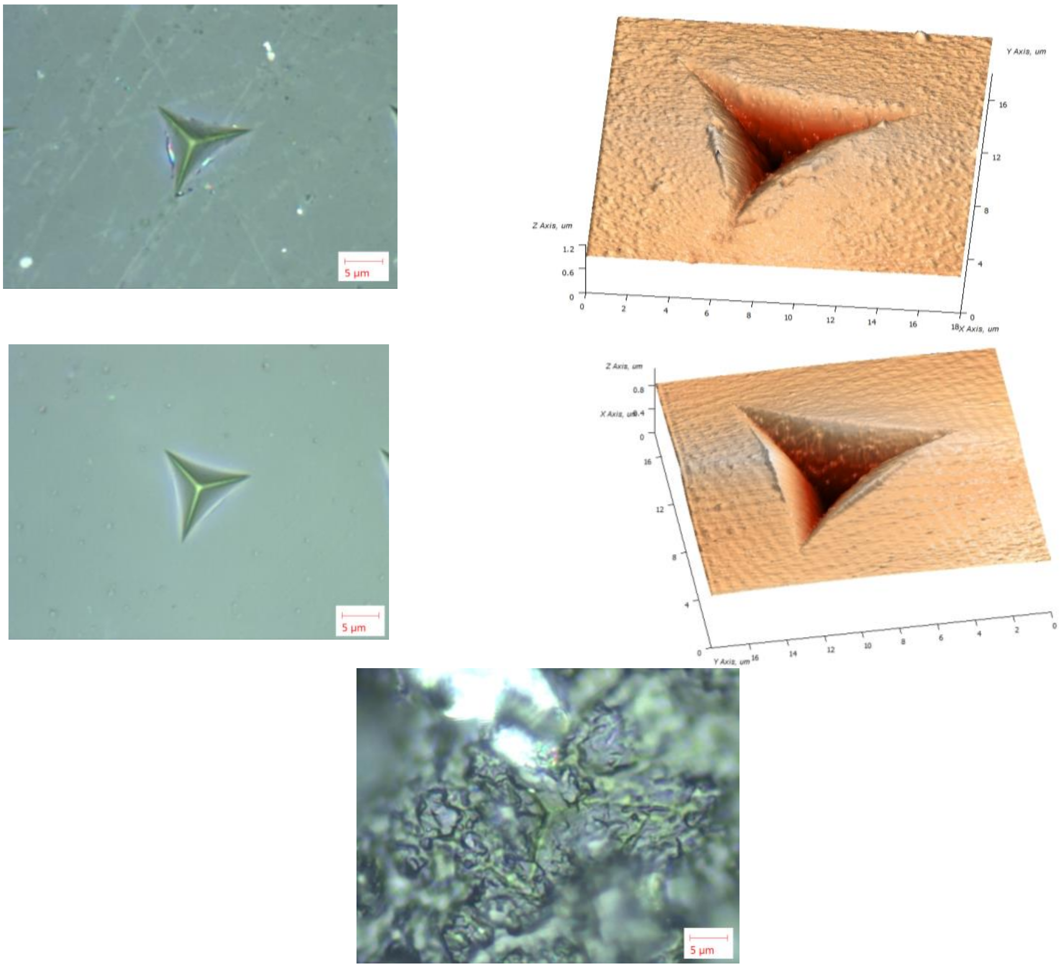

Fig. 3. Indentation imprints of LGC under different finishing procedure analysed by optical (left, x100) and AFM microscopy (normal load of 400 mN) (right): a) polished surface; b) glazed surface; c) grinding surface.

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 may assist in better understanding of the mechanical behaviour of LGC under different finishing procedure and thus facilitate the design and CAD/CAM manufacture for dental restorations.

5. CONCLUSION

The obtained results of nanomechanical properties mostly depend of applied surface finishing procedures. The hardness (HV) and Young's modulus (E) of polished and glazed decreased in a small range when the applied normal load increased. The phenomenon of decreasing HV and E by increasing the indentation load is known under the term "Indentation size effect (ISE)". Grinding sample does not have a trend as polished and glazed. The reason for that is a different surface roughness, the contact between the indenter and surface is realized mostly per tops of roughness.

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.

AFM analysis indicates the occurrence of plastic deformation of the material, as a result of displacement of material (piling-ups) during the penetration of indenter.

6. ACKNOWLEDGMENTS

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