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INFLUENCE OF DIFFERENT FINISHING TECHNIQUES ONTO NANOTRIBOLOGICAL CHARACTERISTICS OF VENEERING GLASS CERAMIC

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Abstract: *This paper describes the nanotribological characterisation of fluorapatite veneering glass ceramic (IPS e.max Ceram, Ivoclar Vivadent) treated with three different surface finishing techniques: polishing, glazing and grinding, using the CSM nanotribometer. Tribological tests was done on the mentioned finishing surfaces against alumina, at nanotribometer by using linear reciprocating module (ball-on-flat), were realised in artificial saliva medium over a range of load 0.25, 0.5, 0.75 and 1N and velocities 4, 8 and 12mm/s. Wear rate was calculated, as the rate of material removal per unit sliding distance, for each conducted test. Also, AFM analysis of different surface finishing procedure was done in order to determine the roughness parameter Ra. The obtained results of nanotribological tests show that all parameters are mostly dependent of different finishing procedure.*

Keywords: *Fluorapatite veneering glass ceramic, Nanotribological characterisation, AFM, Different finishing techniques: polishing, glazing, grinding.*

1. INTRODUCTION

Ceramics have been used as dental materials since the early 18th century [1]. In dentistry, the quest has always been to discover a new restorative material that can precisely replicate the natural structure of teeth [2]. There is a significant need to develop stronger dental ceramics that can perform well in applications involving large amounts of stress, such as crowns and multiple unit restorations [3,4]. With the advancement of science, highly developed dental materials have been created that meet all the requirements of prosthetics, including function, aesthetics, and biocompatibility [2,5]. One of these advanced materials used in aesthetic dentistry is the all-ceramic system.

IPS e.max Ceram is a veneering glass ceramic that produces exceptional aesthetic outcomes when used in combination with all-ceramic systems. It comprises a uniform blend of sintered glass powders and fluorapatite-containing glass ceramic. Veneers, which are thin laminates made of ceramic, are applied to teeth without covering the entire tooth. Veneering ceramics play a crucial role in the appearance of restorations by exhibiting a harmonious interplay of shade, translucency, and brightness that creates a natural and lifelike look [1, 6, 7].

It is crucial to perform tribological characterization of advanced dental materials to fully understand their properties. In the oral cavity, biomechanical functions can cause tribological movements of teeth, restorations, and implants, resulting in wear [8-10]. Wear occurs due to a combination of fundamental processes, including abrasion, adhesion, fatigue, and corrosive effects, which can act synchronously or additively [11-13]. Consequently, the study of the tribology of dental materials has gained increasing attention from various researchers [14].

Also, the tribological properties of ceramic materials are significantly impacted by structural defects and varying surface roughness [1]. For instance, a surface roughness of 0.2 mm is known to create favorable conditions for higher levels of tooth plaque and cavities [15]. However, the smooth surface of intraoral structures provides patient comfort and makes oral hygiene easier [8-10]. Inadequate polishing of the contact surface of a restoration can result in residual surface roughness, which can directly affect the mechanical and aesthetic properties of the contact surface of the material [16-18].

The paper aimed to study the tribological characteristics of fluorapatite veneering glass ceramic (IPS e.max Ceram, Ivoclar Vivadent), under different finishing techniques (polishing, glazing and grinding), using the CSM nanotribometer. The obtained results of tribological measurements were performed at nanotribometer by using linear reciprocating module (ball-on-flat) in artificial saliva medium, and wear rate and wear tracks were presented as obtained results.

2. EXPERIMENTAL PROCEDURE

2.1 Material and samples preparation

IPS e.max Ceram is a type of veneering ceramic used in dental restorations. Its composition is a combination of several components including SiO_2, Li_2O, Na_2O, K_2O, ZnO, and Al_2O_3. The glass structure of the material is reinforced by certain amounts of CaO, P_2O_5, and F, as shown in Figure 1a. These three components are necessary for the formation of a fluorapatite crystal $Ca_5(PO_4)_3F$, which plays a crucial role in the restoration's natural aesthetic appearance in terms of reflection, transparency, and opalescence. The main component of the material's structure is SiO_2, which accounts for about 60% of its weight. Table 1 provides the complete chemical composition of the commercial veneering ceramic IPS e.max Ceram [7,19,20].

Table 1. Chemical composition of Veneering ceramic IPS e.max Ceram [6].

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

a)

b)

Figure 1. IPS e.max Ceram veneering ceramic.

To prepare the samples, molds with a diameter of 20 mm and a height of 5 mm were manufactured. The samples were then sintered at the recommended temperature according to the manufacturer's instructions (Ivoclar Vivadent). After sintering, the contact surfaces of the samples were finished using three different procedures (polishing, glazing, and grinding) as shown in Figure 1b.

For the first sample, diamond sandpaper with varying grits (280, 400, 600, 800, 1200, and 2000) was used to polish the surface under controlled speed, with hand pressure and water cooling. This was followed by fine polishing using a liquid emulsion with a grain size of 6 and 0.04 µm. The second sample was glazed according to the manufacturer’s recommendations (Ivoclar Vivadent). The third sample's contact surface was grinded using a diamond borer (Medin, ISO: 806 314 146 534 016, 150 µm – max) without being strictly fixed.

Finally, before each test, all sample surfaces were cleaned with 70% alcohol to remove any remaining surface contaminants.

2.1 Surface roughness

Surface roughness plays a crucial role in various aspects, such as aesthetics of the contact surface of materials, color changes in dental restoration, secondary caries and gingival irritation, and mutual wear of contact surfaces of teeth and antagonists (natural tooth or dental restoration). In esthetic dentistry, achieving a smooth contact surface finish is the primary goal [1, 19, 21].

Before conducting nanotribological tests, AFM analysis was carried out to determine the roughness parameter R_a. The AFM analysis was performed using NT-MDT manufacturers' equipment located at the Tribology center on the Faculty of Engineering in Kragujevac (Figure 2).

The roughness was measured on all samples within a measurement range of 100x100 µm, and the surfaces' roughness was measured along the same reference length. The results of the roughness parameter Ra obtained from the analysis are presented in Table 2.

Figure 2. NT-MDT Atomic Force Microscopy (AFM).

Table 2. Comparative view of R_a under different finishing techniques of IPS e.max Ceram.

Measuring range, 100x100 µm	**Roughness parameter, R_a**
The polished surface	12.239 nm
The glazed surface	17.253 nm
The grinded surface	0.786 µm

The results indicate that the polished finishing technique has the lowest R_a values, as anticipated. It is important to emphasize that in aesthetic dentistry, the aim has always been to achieve the smoothest possible material contact surface [1].

2.2 Tribological tests

In vitro tribological tests were conducted using a ball-on-flat configuration on the CSM Nanotribometer with a linear reciprocating module, as shown in Figure 3.

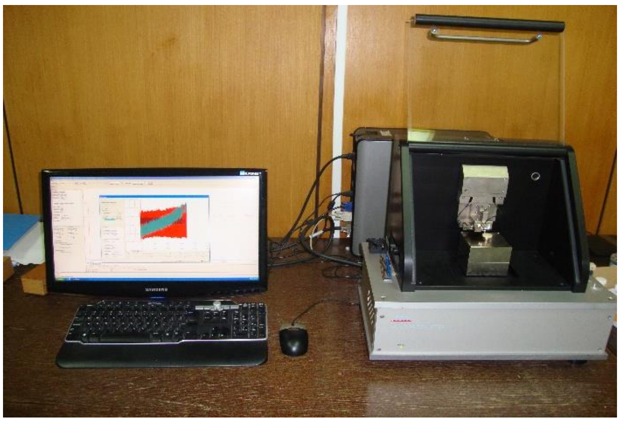


Figure 3. CSM Nanotribometer.

A commercial alumina ball with a diameter of 1.5 mm was used as the static body in contact. The tests were conducted in artificial saliva at room temperature over a range of normal loads (0.25 N, 0.5N, 0.75N and 1N) and sliding speeds (4, 8 and 12 mm/s). Each test lasted for 10,000 fretting cycles, where one cycle represented two full amplitudes of the sliding distance (half amplitude: 0.5 mm; full amplitude: 1 mm). The articulating surfaces were fully immersed in the solution. All tests were repeated three times to ensure reproducibility of the frictional behaviour.

The width and length of wear scars on fluorapatite veneering glass ceramic samples, which were prepared with different finishing techniques (polishing, glazing, and grinding), were observed using optical microscopy (OM) in accordance with ASTM G133-05 [22, 23]. The wear of the alumina ball was not considered in these tests, and was deemed to have "no measurable wear"; only the wear of the flat samples was calculated according to ASTM G133-05.

After each test was completed, the wear volume of the fluorapatite veneering glass ceramic samples was calculated. The wear volume was calculated by using the length of the wear tracks and the average cross-sectional area of the wear track. It was assumed that the cross-sectional area is a flat segment of a sphere corresponding to the geometry of the alumina ball.

3. RESULTS AND DISCUSION

An optical microscope was used to analyze wear tracks, Figure 4 as a representative example (F_n=1 N, V=8 mm/s) displays the optical microscopy of wear tracks of fluorapatite veneering glass ceramic samples that were prepared with different finishing techniques in an artificial saliva medium. These procedures include polishing (Figure 4a), glazing (Figure 4b), and grinding (Figure 4c). It's important to note that at the beginning of the contact, there is point contact between the surface of the sample and the Al_2O_3 ball, but as wear develops, it exceeds into area contact. The magnification of the optical microscope in Figures 4a and 4b is x5, while Figure 4c is x20.

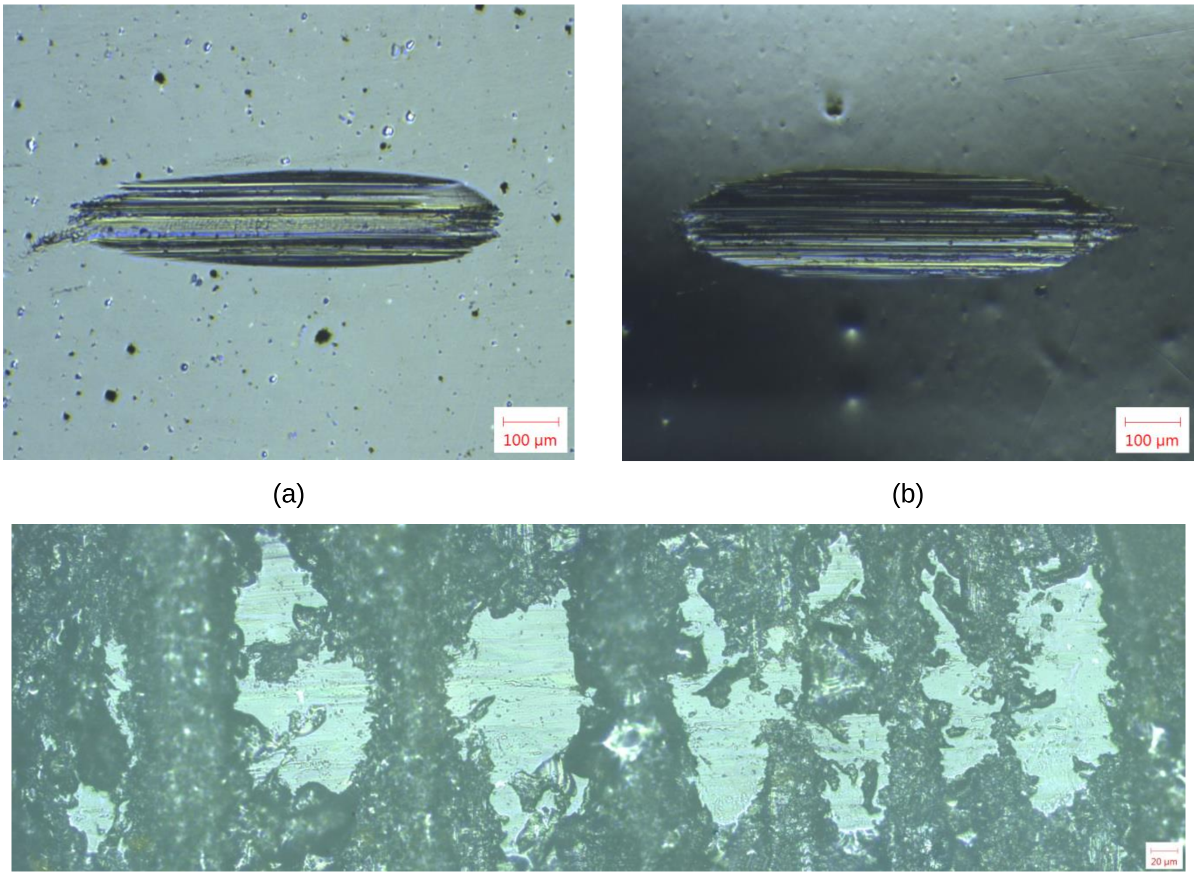
Tribological tests were conducted in an artificial saliva medium with a range of loads of 0.25, 0.5 and 1N, velocities of 4, 8 and 12mm/s, and 10,000 fretting cycles. Figure 4 clearly indicates that different finishing procedures have a significant impact on the shapes of wear tracks. The parallel grooves in the wear tracks of all samples demonstrate a strong correlation between the morphology of the worn fluorapatite veneering glass ceramic surface and the counter body material (Al_2O_3) in the direction of the imposed sliding motion [24].

Table 3 shows the values of the arithmetic mean of the friction coefficients of commercial ceramics IPS e.max Ceram, with different finishing techniques. The numerous values presented in the table indicate that the coefficient of friction varies from ～0.17 to ～0.38, depending on the normal load and sliding speed.

The values of the results clearly show a trend of decreasing values of friction coefficients with an increase in normal load and sliding speed, for all finishing techniques. What is characteristic of this material is that the maximum value of the friction coefficient is ～0.38 and that it occurs at the lowest sliding speed and the lowest normal load. The lowest obtained value of friction coefficient, which involves changing three speeds and four loads, has the grinding. The reason for this is the large accumulation of wear product particles, i.e. impact of a third body in the contact zone.

Miyoshi [25] was the first to report on the influence of tribological parameters on the wear behaviour of dental ceramics. They observed that ceramics behave similarly to metals when they come into contact with the surface of solid bodies. However, for materials that are brittle in nature, the relationship between wear and hardness is not conclusive. In cases where ceramics are in contact with other ceramics or enamel under sliding motion, wear is not caused by plastic deformation as with metals, but rather by microfracture [26].

This form of abrasive wear was introduced by DeLong in 1986 [27]



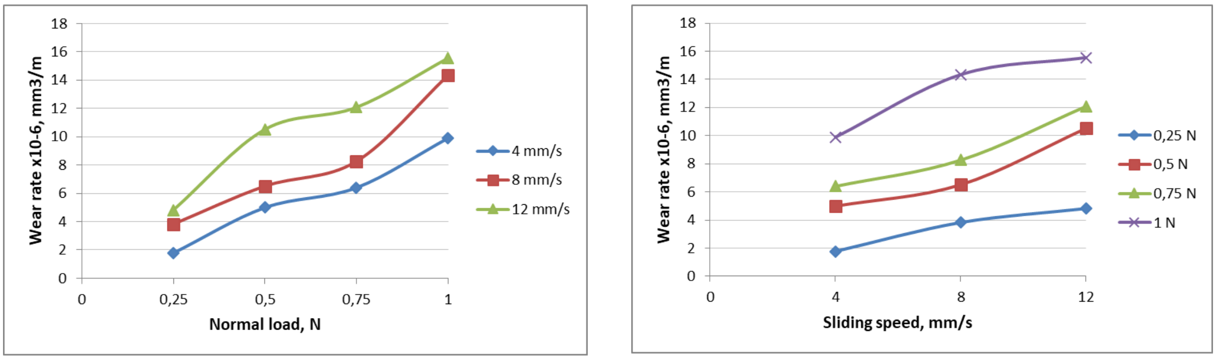
(a) (b)

(c)

Figure 4. Wear tracks of fluorapatite veneering glass ceramic samples prepared with different finishing procedures in artificial saliva medium, F_n=1 N, V=8 mm/s, (a) polished (x5), (b) glazed (x5) and (c) grinding (x20).

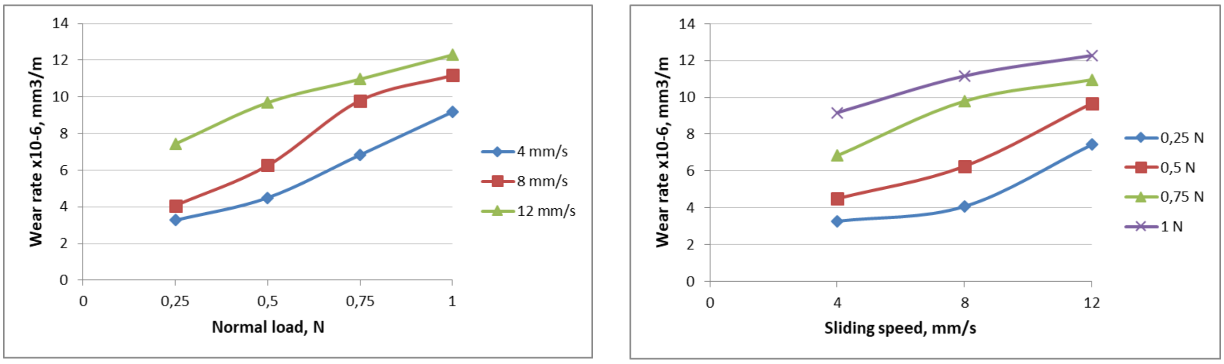
Table 3. Friction coefficient of IPS e.max Ceram under different finishing techniques.

FRICTION COEFFICIENTS				
F_n, N	**v, mm/s**	**Polishing**	**Glazing**	**Grinding**
0.25	**4**	0.387	0.384	0.293
	8	0.364	0.353	0.277
	12	0.362	0.347	0.229
0.5	**4**	0.318	0.294	0.289
	8	0.310	0.274	0.251
	12	0.292	0.247	0.223
0.75	**4**	0.281	0.277	0.261
	8	0.266	0.253	0.241
	12	0.238	0.217	0.176
1	**4**	0.261	0.243	0.258
	8	0.213	0.224	0.189
	12	0.209	0.198	0.175



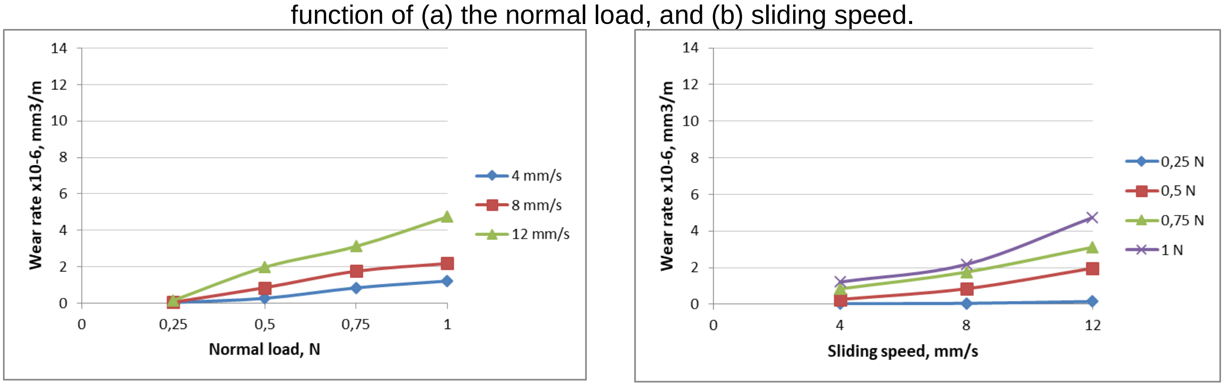
(a) (b)

Figure. 5. Wear rate of polished fluorapatite veneering glass ceramic sample in artificial saliva medium as a function of (a) the normal load and (b) sliding speed.



(a) (b)

Figure 6. Wear rate of glazed fluorapatite veneering glass ceramic sample in artificial saliva medium as a function of (a) the normal load, and (b) sliding speed.



(a) (b)

Figure 7. Wear rate of grinding fluorapatite veneering glass ceramic sample in artificial saliva medium as a function of (a) the normal load, and (b) sliding speed.

Figures 5-7 demonstrate the wear rate of fluorapatite veneering glass ceramic samples (polished, glazed, and grinding) in an artificial saliva medium as a function of normal load and sliding speed.

Polished and glazed surfaces (Figures 5 and 6) exhibit a clear trend of a progressive increase in wear rate with increasing normal load and sliding speed. However, it should be pointed out that the wear rate values of the grinding surface (Figure 7) are significantly lower compared to the previous surfaces (i.e., polished and glazed), with a difference of several times.

Based on the presented optical images of wear tracks (Figures 4a, 4b and 4c), which were obtained under the highest normal load (1 N) and medium sliding speed (8 mm/s) with the presence of artificial saliva, a large number of deep grooves can be clearly observed following the sliding direction distributed uniformly over the entire width of the wear track. These deep grooves are characteristic of abrasive wear that occurs in contact bodies whose hardness significantly differs. In this case, the interacting elements are ceramics against ceramics, with the hardness value of the counter body (Al_2O_3 ball) being significantly higher compared to the hardness values of the tested samples. The wear tracks clearly show that abrasive wear is the dominant wear mechanism. In analyzing dominant wear mechanisms and pronounced abrasion, the influence of wear product particles trapped in the contact zone, as the third body in contact, should also be taken into account.

5. CONCLUSION

The paper presents nanotribological characterisation of fluorapatite veneering glass ceramic prepared with different finishing techniques (polishing, glazing and grinding). Attained results show that:

- The values of friction coefficients show a trend of decreasing values of friction coefficients with an increase in normal load and sliding speed, for all finishing techniques.
- The wear rate increases gradually within a narrow range as the normal load is increased, regardless of the sliding speed.
- Abrasive wear was found to be the dominant wear mechanism in all samples examined.
- The wear tracks' shapes and wear rate values were significantly affected by different finishing techniques applied to the samples.

The results that have been presented could aid in improving the understanding of the nanotribological characteristics of dental ceramics made from fluorapatite veneering glass ceramic when subjected to various finishing techniques. As a result, this could simplify the process of designing, selecting, and using CAD/CAM technology for the manufacture of dental restorations.

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