Serbian Tribology Society

SERBIATRIB '17

15th International Conference on Tribology

Faculty of Engineering
University of Kragujevac

Kragujevac, Serbia, 17 – 19 May 2017

MICRO SCRATCH BEHAVIOUR OF LITHIUM DISILICATE GLASS CERAMIC AFTER GLAZING TREATMENT

Marko PANTIĆ[1], Slobodan MITROVIĆ[1], Miroslav BABIĆ[1], Danimir JEVREMOVIĆ[2],
Tatjana V. KANJEVAC[3], D. DŽUNIĆ[1*], D. ADAMOVIĆ[1]
[1]University of Kragujevac, Faculty of Engineering, Kragujevac, Serbia
[2]University Business Academy, School of Dentistry, Pančevo, Serbia
[3]University of Kragujevac, Faculty of Medical Sciences, Department of Dentistry, Kragujevac, Serbia
*Corresponding author: dzuna@kg.ac.rs

Abstract: *The purpose of this in vitro study was to investigate micro scratch behaviour of lithium disilicate glass ceramics (LDGC) - IPS e.max CAD, after glazing treatment. LDGC is one of most used all-ceramic dental system which provides high aesthetic quality and all patient requirements: function, biocompatibility and aesthetic. Micro-scratch test was performed with a progressive load of 0.01 to 30 N, which meant that the values of the normal force increased with the change of time. Rockwell C diamond cone was used as the indenter, of radius tip 100 µm. The aim of this study is to test the wear resistance of glazed lithium disilicate, to determine the thickness of the glaze and quantify the tribo-mechanical characteristics of the glazed layer. The obtained results are presented in the form of values of the critical loads (L_c), the friction coefficient (µ), the penetration depth (P_d) and the residual depth (R_d).*

Keywords: *Micro Scratch Test, Lithium disilicate glass ceramic, Glazing tretments, Tribo-mechanical properties*

1. INTRODUCTION

In recent years, glass ceramics has become very important and one of the most commonly used materials in prosthetics, solely due to its excellent aesthetic characteristics, good mechanical strength and durability of restorations.

In 1991 the company Ivoclar Vivadent with IPS Empress System had the first breakthrough in the development of glass ceramics in aesthetic dentistry. Further development of glass ceramics was achieved in 1998 with the emergence of IPS Empress 2 system which was based on lithium disilicate [1]. The material is characterized by the high values of flexural strength and by the wide application in making restorations and bridges. A new advanced all-ceramic system called IPS e.max has been developed (Ivoclar Vivadent, Liechtenstein), based on the idea that the future of all-ceramic systems lies in the use of PRESS and CAD/CAM technology. It contains a superior aesthetic and high-resistant materials intended for the PRESS and CAD/CAM technology. IPS e.max system covers a wide range of indications of ceramic restorations in different zones of the load, combining a large flexural strength, aesthetics and ease of manufacture [2].

A large number of publications in the field of dentistry, and the fields that are close to it, confirm the use of scratch test for a number of tribological testing of various biomaterials [3-10]. The ability of scratch test is to mechanically characterize the surface layer of the material that is being tested and to quantify the parameters such as the friction and the adhesion forces. It is widely used during the testing of all types of thin coatings and films (thickness of 0.1 - 30 µm), which forms nowadays the essential tool for the research, development and quality control of the surface layers of materials.

It is generally known that the scratch test is used to analyse all types of thin coatings and films. In aesthetic dentistry, the purpose of the glaze, as the form of the coating, is to reduce the porosity and surface roughness of the material itself and to improve the aesthetic appearance of restorations in the form of an aesthetic shine [11-15]. The important thing that must be emphasized is that the glaze with its presence on the surface layer of the ceramic does not improve the strength of the self-material [16].

The aim of this study is to test the wear resistance of glazed lithium disilicate, to determine the thickness of the glaze and quantify the tribo-mechanical characteristics of the glazed layer. The obtained results are presented in the form of values of the critical loads (L_c), the friction coefficient (μ), the penetration depth (P_d) and the residual depth (R_d).

2. EXPERIMENTAL PROCEDURE

MST Anton Paar micro-scratch tester, located at the Tribology center on the Faculty of Engineering in Kragujevac, was used to determine the tribo-mechanical characteristics of the glazed lithium disilicate (Figure 1).

The working principle of micro-scratch device is shown in Figure 2. The indenter tip (Rockwell C diamond cone, radius of the tip 100 µm) passes over the surface layer of the material with constant and progressive load, depending on the protocols defined in the software. The sample is fixed in the sample holder which is located on the anti-vibration table and moves at a constant speed on the X and Y axes. Normal force, penetration depth, acoustic emission and friction force are detected during the testing. Acoustic signal detector is placed above the diamond needle, and it registers the vibrations which appear when damage occurs. In addition to the acoustic sensor, the scratch test device has also encoders that register the friction force. The software may provide real-time results of the measurement. When the indenter moves on the sample, it comes to formation of various damages depending on the value of the applied force. The minimum value of the normal force (F_n) that causes damage is referred to as a critical load (L_c). Three values of critical load are most commonly detected (L_{c1}, L_{c2} and L_{c3}).

Figure 1. MST Anton Paar micro-scratch tester

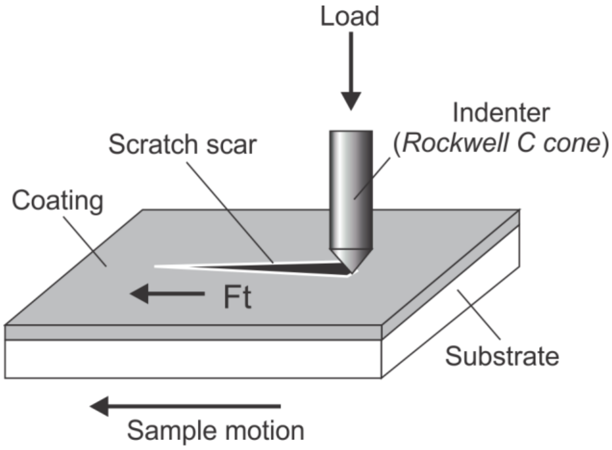

Figure 2. The schematic view of scratch test and the working principle

Rockwell C diamond cone was used as the indenter, of radius tip 100 µm. The test was performed with a progressive load of 0.01 to

30 N, which meant that the values of the normal force increased with the change of time. The range was chosen as widely as possible in order to monitor the reaction of fully glazed surface on the change of load. Sliding speed of the sample, which was fixed in the sample holder on the work table, was constant 1 mm/min. The defined length of the scratch scar was 3 mm. The experiment was performed in the conditions without lubrication in ambient temperature of 23 ±2°C and the test was repeated three times. Before the testing, the glazed sample was ultrasonically cleaned well (30 minutes) and afterwards it was cleaned with 70 % alcohol in order to remove all the surface contaminants.

2.1 Material and samples preparation

IPS e.max CAD is the type of all-ceramics, which is characterised by a high aesthetic quality, so it can be said that its characteristics meet all prosthetic requirements in form of: aesthetics, function and biocompatibility of the material [15]. Table 2.4 presents the chemical composition of the commercial lithium disilicate IPS e.max CAD. SiO_2 and LiO_2 represent components which form $Li_2Si_2O_5$ crystals, while the P_2O_5 is added as a nucleating agent. Other Oxides with its characteristics further contribute to the material structure itself.

Table 2.4 Chemical composition of IPS e.max CAD [17]

Standard composition	(in % by weight)
SiO_2	57.0 – 80.0
Li_2O	11.0 – 19.0
K_2O	0.0 – 13.0
P_2O_5	0.0 – 11.0
ZrO_2	0.0 – 8.0
ZnO	0.0 – 8.0
Al_2O_3	0.0 – 5.0
MgO	0.0 – 5.0
Colouring oxides	0.0 – 8.0

The glazed sample is in the form of a block, the length of 18 mm, a width of 14 mm and a height of 12 mm. Before the glazing process, lithium disilicate is crystalized at the prescribed temperature according to instructions of the manufacturer Ivoclar Vivadent. After the crystallization, the surface of the sample is glazed as the form of the final treatment of the material, also according to the exact prescribed procedure of the manufacturer Ivoclar Vivadent. The glaze is applied by hand with a brush in a thin layer on a contact surface of the sample and represents the type of protective coating, wherein it is baked afterwards in an oven at the accurately prescribed temperature.

3. RESULTS AND DISCUSION

Tribo-mechanical tests were preceded by the AFM analysis in order to determine the roughness parameters Ra, 3D topography and surface roughness profile of the tested material (Figure 3).

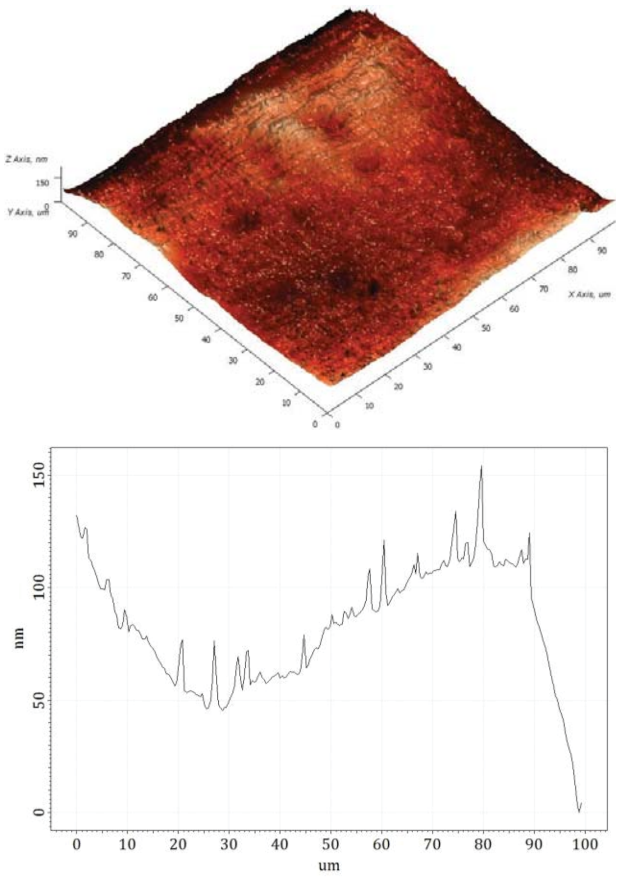

Figure 3. AFM analysis of IPS e.max CAD glazed surface - 3D topography and surface roughness profile

The measured value of the surface roughness (Ra) of the lithium disilicate glazed surface (IPS e.max CAD) is Ra = 19.817 nm. It is known that the process of glazing, due to uneven application of the glaze layer, leads to surface irregularities in the form of wavy surfaces and gas bubbles, which can be clearly seen on the 3D image of the surface topography.

During the experimental testing, first the scanning (Pre-scan) of the contact surface was performed at the place of testing, due to the imperfections of the glazed surface (wavy surfaces), as well as different surface roughness, which oscillates at a micro/nano-level. Due to the penetration of the MST indenter in the surface layer of the glaze, at progressive load and movement of the sample at a sliding speed of 1 mm/min, there is a gradual formation of scratch scar of a certain depth. Due to the elastic relaxation of the material/coating itself, the new analysis of the surface was performed in the scratch scar itself after the end of scratch test (Post-scan) to determine the actual residual depth (R_d) in relation to the initial measured penetration depth (P_d). The obtained values of the different penetration depths, as well as the the friction coefficient, represent the mean values of 3 measurements and they entirely define the tribo-mechanical properties of the glazed surface of the material.

Figure 4 shows the scratch scar on the glazed surface of the material that comparatively follows the diagrams of the dependence of different penetration depths (P_d and R_d) depending on the friction coefficient and normal load. The loose particles of the surface layer of the glaze around the scratch scar, of different sizes, could be easily seen in the Figure 4, and they are made as a result of indentation and movement of Rockwell C diamond cone at the surface itself. The mechanisms that occur during the scratch test are usually micro-cutting and micro-ploughing. The panoramic view of the scratch scar was formed on the optical microscope which is an integral part of the device, with a magnification of 5×. Black spots that represent the surface imperfections of finial treatment in the form of wavy surfaces and gas bubbles, which are characteristic for the glazing treatment, can be seen in some optical panoramas of scratch scars. The Figure 4 clearly shows the trend of increase of friction coefficient values with a slight oscillation along the sliding distance, due to the increase of the normal load. The variation of the friction coefficient which appears in the form of distinct jumps and falls at the diagrams largely follows the trend of the oscillation of the value of the penetration depth (P_d) and residual depth (R_d), which clearly defines their mutual dependence.

The maximum value of the friction coefficient of lithium disilicate is ~0.22 and it penetration depth of the material of 34 mm, due to the effect of normal load of ~27 N. The residual depth (R_d) in relation to the measured penetration depth (P_d) is 15 mm. The value of the thickness of the glazed layer, read from the diagram R_d, is 20 µm.

Regarding the measured values of the critical loads, due to which there is an abrasive removal of the glazed layer at the progressive increase of normal load, Figure 5 shows their different critical stages in the scratch scar itself. Panoramic views of all scratch scars were photographed by OM with 5× magnification, while the individual images of the critical stage of the scratch scars, are presented with a magnification of 20×.

The abrasive wear, as a dominant type of wear, can be clearly seen in shown Figures 5 of the scratch scars. The reason is a drastic difference in the hardness of the two contact bodies during the relative motion. Rockwell diamond indenter has a significantly higher hardness in comparison to the hardness value of the tested sample.

Figure 5, characterised by different phase damages of the surface glazed layer of lithium disilicate, clearly show that the initial defects occur at normal load of the indenter of 4.5 N (L_{C1}). During the progressive increase of the normal load, the penetration of glazed surface occurs in the moment when the normal load reaches a value of ~18 N.

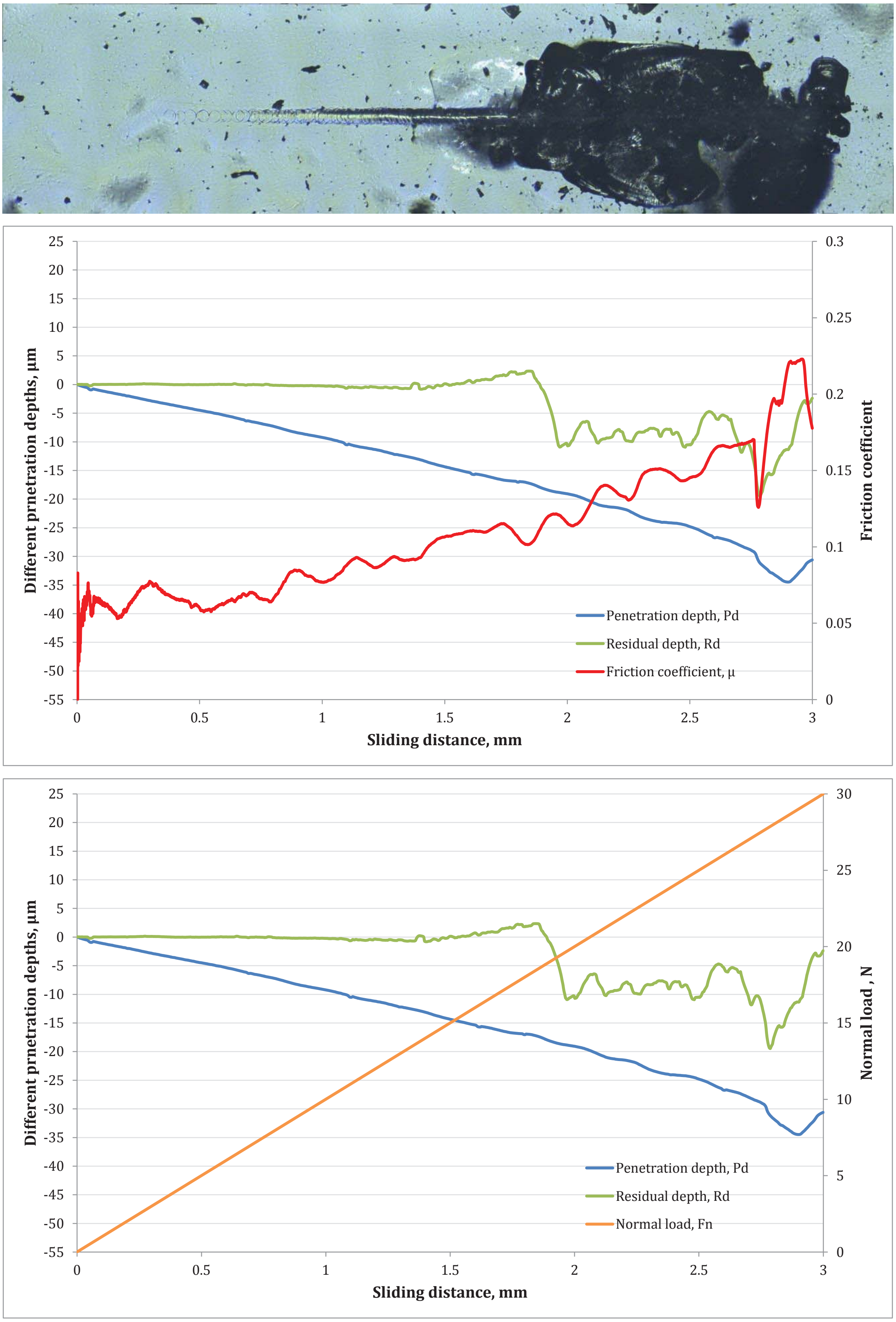

Figure 4. The scratch scar display of the glazed surface of lithium disilicate followed by the diagrams of different penetrating depths (P_d, R_d) depending on the friction coefficient and normal load

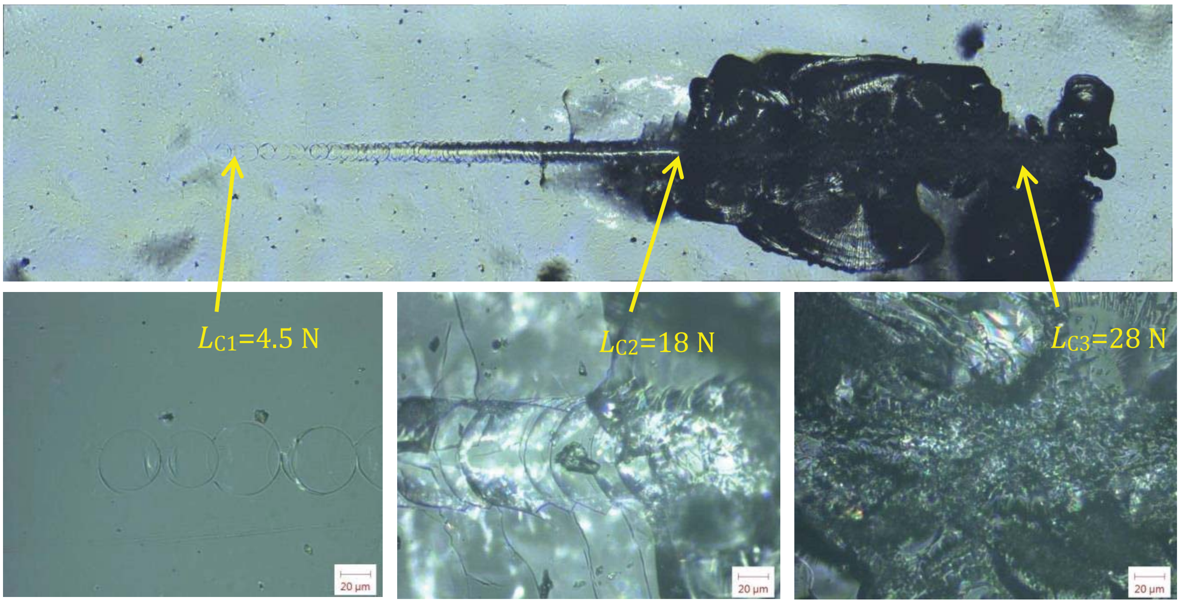

Figure 5. Defining critical loads due to which there is a different phase damages of the glazed lithium disilicate

This coating penetration can be clearly seen on the optical display L_{C2}, with magnification of 20×. After that moment, the indenter comes in contact with the surface of the base material. The further increase of the value of normal load, at the value of 28 N, shall cause the surface damages of lithium disilicate and encroachment of the indenter into the base material, which is clearly seen on the third image for L_{C3} value. All values of critical loads are obtained as mean values of three repeated measurements of the scratch test by statistical analysis in the software of MST. Also, all these changes of the critical loads are followed by the increase of the friction coefficient (μ) and the penetration depth (P_d and R_d), whose analysis are previously discussed.

The obtained results of this *in vitro* studies represent a very useful and unique information for the future comparison with clinical *in vivo* studies of the similar type, because at the time of literature review, there was no paper found with the similar subject and test plan relating to the tribo-mechanical tests of the glazed surfaces of the all-ceramic systems.

4. CONCLUSION

Scratch test was performed in order to determine the tribo-mechanical characteristics of glazed surfaces of commercial lithium disilicate ceramics. The optical display of the scratch scar shows that the glazed layer is very brittle. The AFM analysis has confirmed that the glaze surface is very wavy, which means that the glaze thickness itself may vary depending on the location of test. The experiment has confirmed that the thickness of the glazed layer based on the residual depth is about 20 µm.

Based on the obtained tribo-mechanical characteristics of glazed layer, their mutual dependence can be easily stated or that the changes of the friction coefficient values over time are in direct correlation with changes of the values of penetration depth (P_d) and residual depth (R_d).

A damage of the glazed layer occurs in the three characteristic phases during the progressive increase of the normal load. The effect of the force of 4.5 N causes the initial damage of glazed layer, and the moment of the glaze penetration during the abrasive effect of the indenter occurs at the indenter force of 18 N, while the total delamination of the glazed layer, in combination with the surface damages of the base material, occurs at the force value of 28 N.

Based on the all presented in the paper, the presented results of scratch analysis represent an excellent basis for all future tribo-mechanical tests of a similar type, as well as the possibility of more detailed analysis of the present phenomena by using SEM microscopy.

ACKNOWLEDGEMENT

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

REFERENCES

[1] ASTM C1161 *Standard Test Methods for Flexural Strength of Advanced Ceramics at Ambient Temperature*, 2002.

[2] M. Pantić: *Tribological characterization of advanced dental materials*, PhD thesis, University of Kragujevac, Faculty of Engineering, Kragujevac, 2017.

[3] L.A. Flanders, J.B. Quinn, O.C. Jr Wilson, I.K. Lloyd: Scratch hardness and chipping of dental ceramics under different environments, Dent Mater, vol. 19, no. 8, pp. 716-724, 2003.

[4] S. Fouquet, M. Rollin, R. Pailler, X. Bourrat: Tribological behaviour of composites made of carbon fibres and ceramic matrix in the Si–C system, Wear, vol. 264, pp. 850–856, 2008.

[5] M.S. Al-Haik, S. Trinkle, D. Garcia, F. Yang, U. Martinez, H. Sumali, S. Miltenberger: Investigation of the nanomechanical and tribological properties of dental materials, Int. J. Theoretical and Applied Multiscale Mechanics, vol. 1, no. 1, pp. 1-15, 2009.

[6] A. Vencl, S. Arostegui, G. Favaro, F. Živić, M. Mrdak, S. Mitrović, V. Popović: Evaluation of adhesion/cohesion bond strenght of the thick plasma spray coatings by scratch testing on coatings cross-sections, Tribology International, vol. 44, no. 11, pp. 1281-1288, 2011.

[7] F. Živić, M. Babić, D. Adamović, S. Mitrović, P. Todorović, G. Favaro, M. Pantić: Influence of the surface roughness on adhesion of chrome coatings on alloy tool steel X165CrMoV12, Journal of the Balkan Tribological Association, vol. 18, no. 2, pp. 228–237, 2012.

[8] J. Zheng, Y. Li, M.Y. Shi, Y.F. Zhang, L.M. Qian, Z.R. Zhou: Micro tribological behaviour of human tooth enamel and artificial hydroxyapatite, Tribology International, vol. 63, pp. 177–185, 2013.

[9] S. Palaniappan, J.-P. Celis, B.V. Meerbeek, M. Peumanas, P. Lambrechts: Correlating in vitro scratch test with in vivo contact free occlusal area wear of contemporary dental composites, Dental Materials, vol. 29, pp. 259-268, 2013.

[10] O. Borrero-Lopez, A. Pajares, P.J. Constantino, B.R. Lawn: Mechanics of microwear traces in tooth enamel, Acta Biomaterialia, vol. 14, pp. 146-153, 2015.

[11] K.C. Cheung, B.W. Darvell: Sintering of dental porcelain: effect of time and temperature on appearance and porosity, Dent Mater J., vol. 18, pp. 163-173, 2002.

[12] G. Aksoy, H. Polat, M. Polat, G. Coskun: Effect of various treatment and glazing (coating) techniques on the roughness and wettability of ceramic dental restorative surfaces, Colloids Surf B Biointerfaces, vol. 53, no. 2, pp. 254-259, 2006.

[13] K. Mehulic, V. Svetlicic, S. Segota, D. Vojvodic, I. Kovacic, D. Katanec, N. Petricevic, D. Glavina, A. Celebic: A Study of the Surface Topography and Roughness of Glazed and Unglazed Feldspathic Ceramics, Coll. Antropol, vol. 34, pp. 235–238, 2010.

[14] S. Štefančić, L. Ćurković, G. Baršić, M. Majić-Renjo, K. Mehulić: Investigation of Glazed Y-TZP Dental Ceramics Corrosion by Surface Roughness Measurement, Acta stomatol Croat., vol. 47, no. 2, pp. 163-168, 2013.

[15] M. Pantić, S. Mitrović, M. Babić, D. Jevremović, T. Kanjevac, D. Džunić and D. Adamović: AFM Surface Roughness and Topography Analysis of Lithium Disilicate Glass Ceramic, Tribology in Industry, vol. 37, no. 4, pp. 391-399, 2015.

[16] T. Asai, R. Kazama, M. Fukushima, T. Okiji: Effect of overglazed and polished surface finishes on the compressive fracture strength of machinable ceramic materials, Dent Mater J., vol. 29, no. 6, pp. 661-667, 2010.

[17] K. Fischer, P. Bühler-Zemp, T. Völkel: *Scientific Documentation IPS e.max CAD*, Ivoclar-Vivadent AG, Schaan, 2011.