SURFACE AND DEPTH MODIFICATION ASSESSMENT FOR BIOMATERIALS USED IN RESTORATIVE DENTISTRY

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Implementation of a large number of materials into restorative dentistry practice, materials which have a variable composition, often puts us in difficulty in choosing one of them able to have the qualities adapted to a given clinical situation. This is because, every material class requires its own technology for processing and finishing, which, if is not correctly followed, may compromise the benefits and the performance of the material. Structural changes or surface appearance of materials, which are variations arising both from technological processes and during use, not only adversely affect the mechanical strength of restorative structures, but impugn their biological qualities under the influence of oral environmental conditions. For these reasons, this paper will present experimental results on the physico-chemical and geometrical evaluation of surface after wear process produced by abrasion. The researches aim the assessing of the surfaces quality through their geometry based on profilometry and atomic force microscopy.

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1. Introduction

The surface roughness of ceramic material determines the increase of plaque accumulation affecting the quality of restorations. It is known that a greater roughness than 0,2 mm determines favorable conditions for a higher occurance of plaque accompanied by a high risk for dental caries or periodontal disease.

Clinical experience, otherwise attract attention to possible side effects in the presence of restorative materials with doubtful physical qualities (metal alloy and acrylic materials, glass ionomers cements, etc.), which creates difficulties in identifying stakeholders.

Humidity, temperature and pH variations for oral cavity may contribute to changes of the restorative materials surfaces, particularly of roughness appearance which is favorable for the formation of ecological niches that allow growth of microorganisms, which are protected by applied hygiene measures and frictional forces.

Because the variations in pH of oral environment, especially for acidity, have the ability to affect the material surface, wear resistance, absorption capacity and solubility; analysis of changes in solutions with different pH values is important for future measures able to improve the quality of materials used in dentistry.

The surface qualities of restorative materials in terms of functional and biological applications of oral environment, are based both on some of the material qualities from which they are made and on their processing technology, which is not always an optimal one.

A main importance in the study of dental materials and alloys reveal the possibility to introduce

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specific technologies that do not strongly influence their primary structure and surface quality, influencing in these conditions the passivation in oral environment.

The resistance qualities under functional and para-functional requirements, under the action of chemical or biological agents with an intensive corrosive action are influenced on the one hand by the structural quality of the material and on the other hand by their specific technological process.

The resulted changes in the mass of restorative materials after wear process can affect the both surface layers and deeper layers accompanied by a negative influence both on the mechanical strength and surface characteristics with a main direct or indirect impact on neighboring tissues.

Geometric quality assessment of resulted surfaces after the wear process based only on the roughness appreciation does not provide an overview of it. The roughness with the same average height can take different forms (eg flat or sharp), forms that can be identified microscopically and which allow a different behavior of surface. For these reasons, it is appropriate to use parameters which provide information on slope, curvature, wavelength and their distribution. It is known that an increased surface roughness leads, through established mechanisms, to a concentration of stress accompanied by the crack initiation in the mass of material and, in main cases, a strong accelerated propagation of them.

The texture of measurable surface can support the understanding of material wear phenomena. It also may indicate that the roughness height represents only an estimation of the surface quality; the aspect of horizontal roughness is often not taken into account when it is used for the determination parameter called *roughness*.

2. Materials and maethods

The surface quality after its wear process was apprecieted with the help of roughness profile specific for four ceramic materials: *D.SIGN* (IVOCLAR), *IN LINE* (IVOCLAR), *HERA-CERAM* (Heraeus Kulzer) şi *VM13* (Vita-Vident TN) - figure 1.

The surface morphology resulted after wear process was investigated by atomic force microscopy based on APCA-2400 microscope type.

Fig. 1. Samples of used ceramic materiale.

There were obtained two-dimensional and three-dimensional topographic images over an analyzed area of 10 x 10 mm with a resolution of 256 pixels. Based on this method it can be possible to determine force variations by 10^{-9} N order.

For the study of roughness parameters over a greater area greater it was used a Surtronic roughness device made by Taylor-Hobson Ltd. (England), using five steps for each calculation of roughness, and recording three routes for each sample in three different locations (perpendicular , oblique, parallel) related to studied surface. Their average was used as the final result for the interpretation of size and shape for each sample.

Measurements were performed at Faculty of Physics, Department of Physics of Solids - "Al. I. Cuza " University of Iasi - Romania and Faculty of Mechanics, Department of Machine Elements and Tribology - Polytechnic University of Bucharest - Romania.

3. Results and discussion

Topographic features of the used surface compared with unused surfaces obtained through atomic force microscopy techniques are presented in below tables.

Table 1. Topographical characteristics of surfaces for IN LINE material

	Topographical characteristics (nm)	Used surfaces	Unused surfaces
1 x1	Average height	- 0,18 nm	0,08 nm
	Ra roughness	7,17 nm	2,03 nm
	RMS roughness	9,04 nm	3,08 nm
30 x 30	Average height	0,002 µm	0,3 nm
	Ra roughness	0,128 µm	34,0 nm
	RMS roughness	0, 168 µm	45,7 nm
5 x 5	Average height	0,8 nm	- 0,3 nm
	Ra roughness	29,7 nm	10,3 nm
	RMS roughness	38,7 nm	13,0 nm

It is found that the roughness of studied surfaces increases with friction length. If for *IN LINE* ceramic material the experimental results indicate a relatively smooth surface with an initial low roughness Ra of about 7,17 nm and a height of roughness RMS (root-mean square roughness) of 0,168 µm, for *D.SING* material average height of profile is not much different from the previous one (8,02 nm, 30,7 nm respectively), but its roughness is closed as value by the roughness considered as optimal at the oral level (Table 1.)

Variation in mean square roughness of this surface layers is shown in 3D images of atomic force microscopy performed in the area of used *IN LINE* studied material - figure 2.

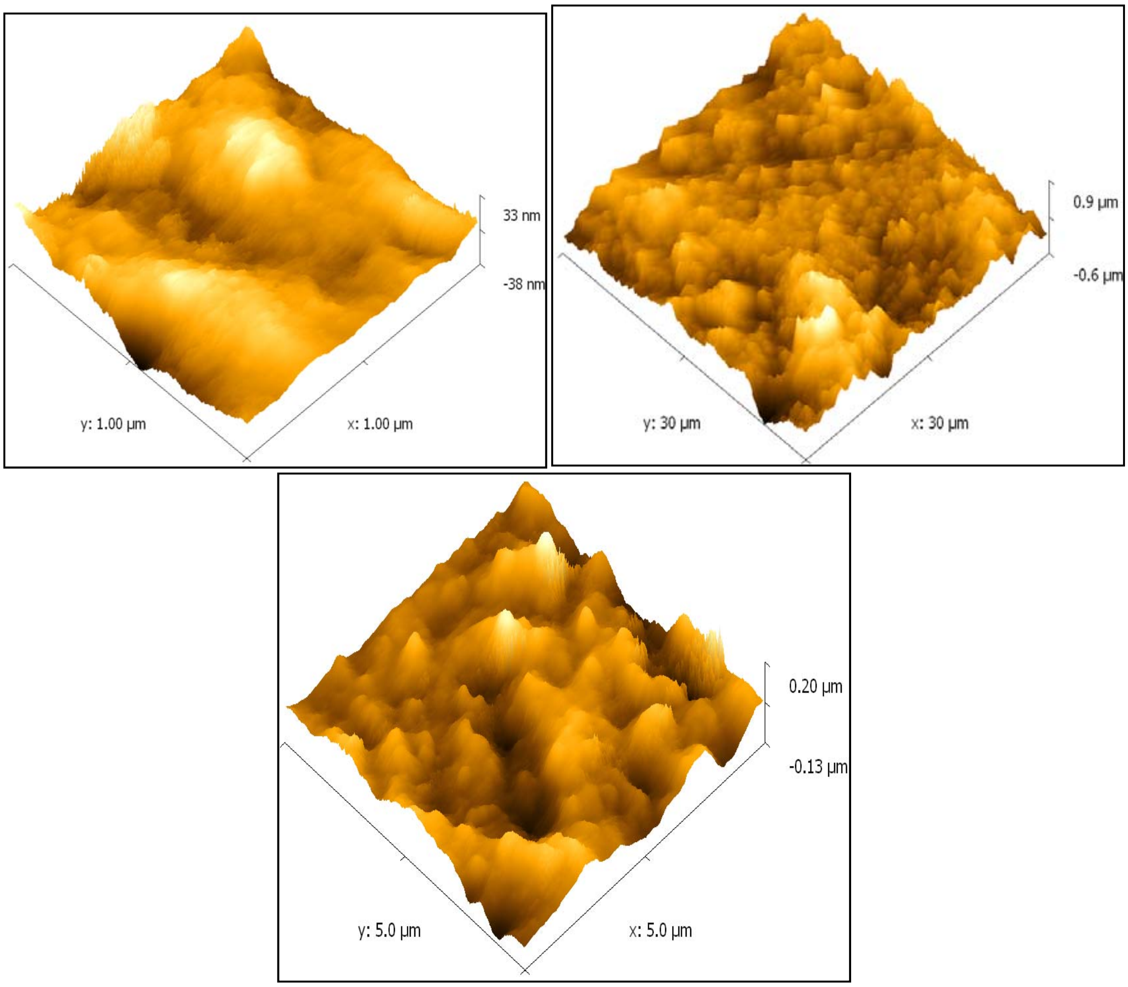

Fig. 2. Atomic force microscopy images at used surface level for IN LINE ceramic material

For comparison, in figure 3 are presented 3D images for unused surfaces of *IN LINE* ceramic material, taken as a witness in this trial.

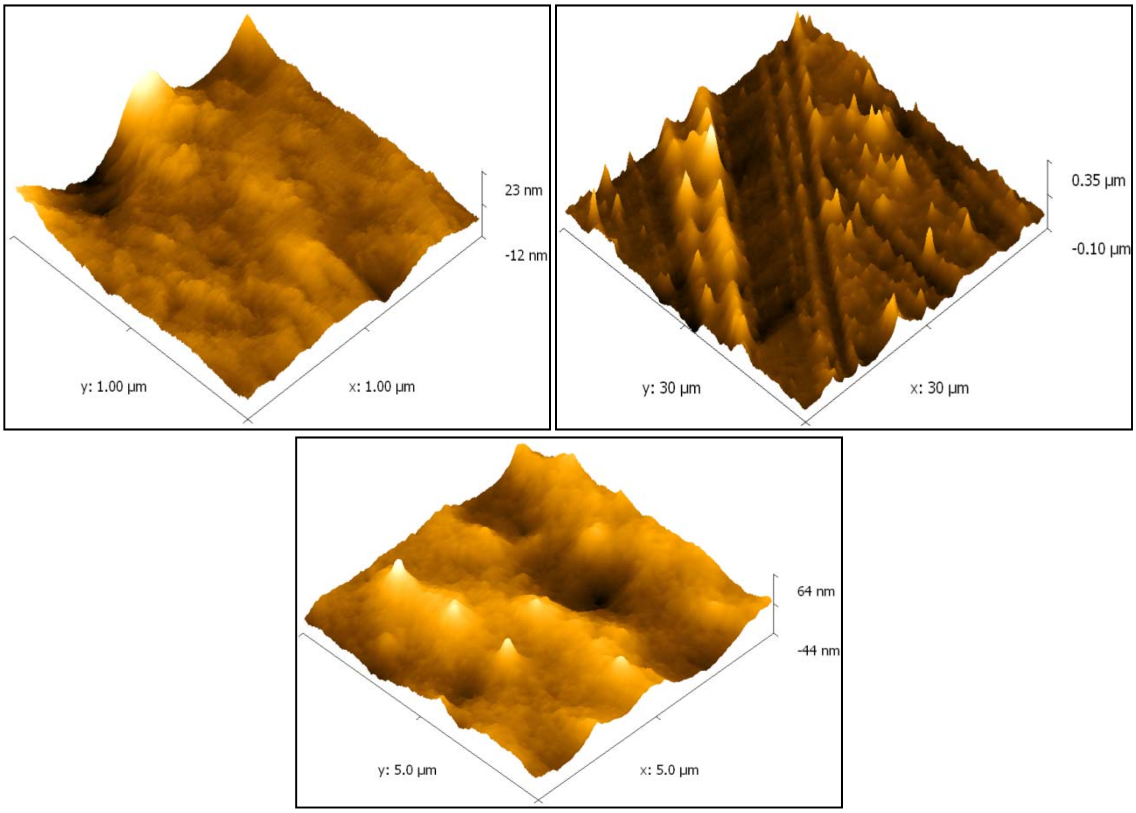

Fig. 3. Atomic force microscopy images at unused surface level for IN LINE ceramic material

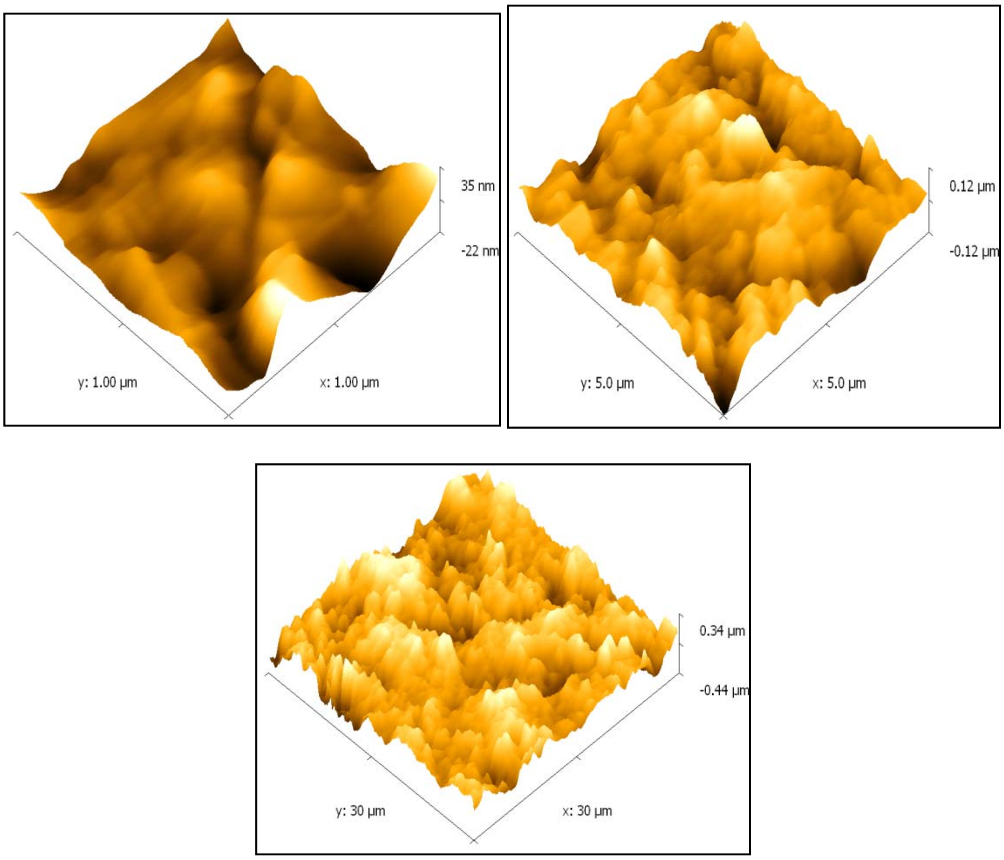

Fig. 4. Atomic force microscopy images at used surface level for D.SING ceramic material

For *D.SING* material is visible a superior quality of the exposed surfaces compared to the other studied ceramic materials before and after tests on abrasion wear behavior; the obtained values are placing the roughness in optimum perimeter for prosthesis.

The effect of wear on the *D.SING* ceramic surface is shown in Fig. 4, while in figure 5 is shown the 3D aspect of unused surface.

As can be seen from figure 4, a major influence of load increasing depending on the length of friction on studied surface topology is manifested in the appearance of ceramic surface layer.

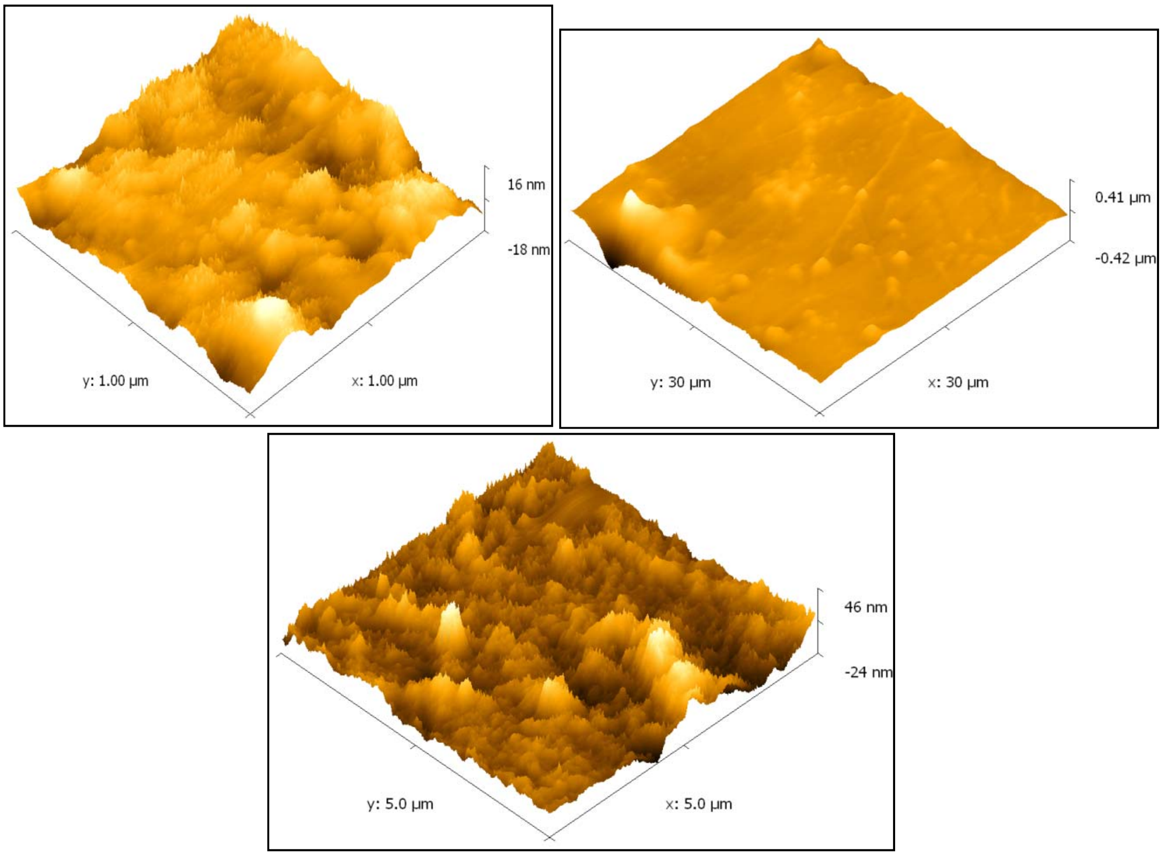

Fig. 5. Atomic force microscopy images at unused surface level for D.SING ceramic material

Table 2 shows the results for roughness before and after aging process. Relatively high hardness of this type of ceramic material is reflected in the relatively small changes in surface quality achieved by simulating an abrasion wear process.

Table 2. Topographical characteristics of surfaces for D.SING material.

	Topographical characteristics (nm)	Used surfaces	Unused surfaces
1 x 1	Average height	0,06 nm	- 0,02 nm
	Ra roughness	2,67 nm	5,86 nm
	RMS roughness	3,52 nm	8,02 nm
30 x 30	Average height	0,8 nm	1,5 nm
	Ra roughness	24,2 nm	82,3 nm
	RMS roughness	41,5 nm	104,8 nm
5 x 5	Average height	0,11 nm	0,1 nm
	Ra roughness	5,42 nm	24,2 nm
	RMS roughness	7,50 nm	30,7 nm

For *HERA-CERAM* material the roughness gradually increases to 9,0 nm and reaches a maximum of 0,156 µm; the reduction of load determines a decrease of Ra roughness and RMS height (reaching 41,7 nm, 53,1 nm respectively) figure 6

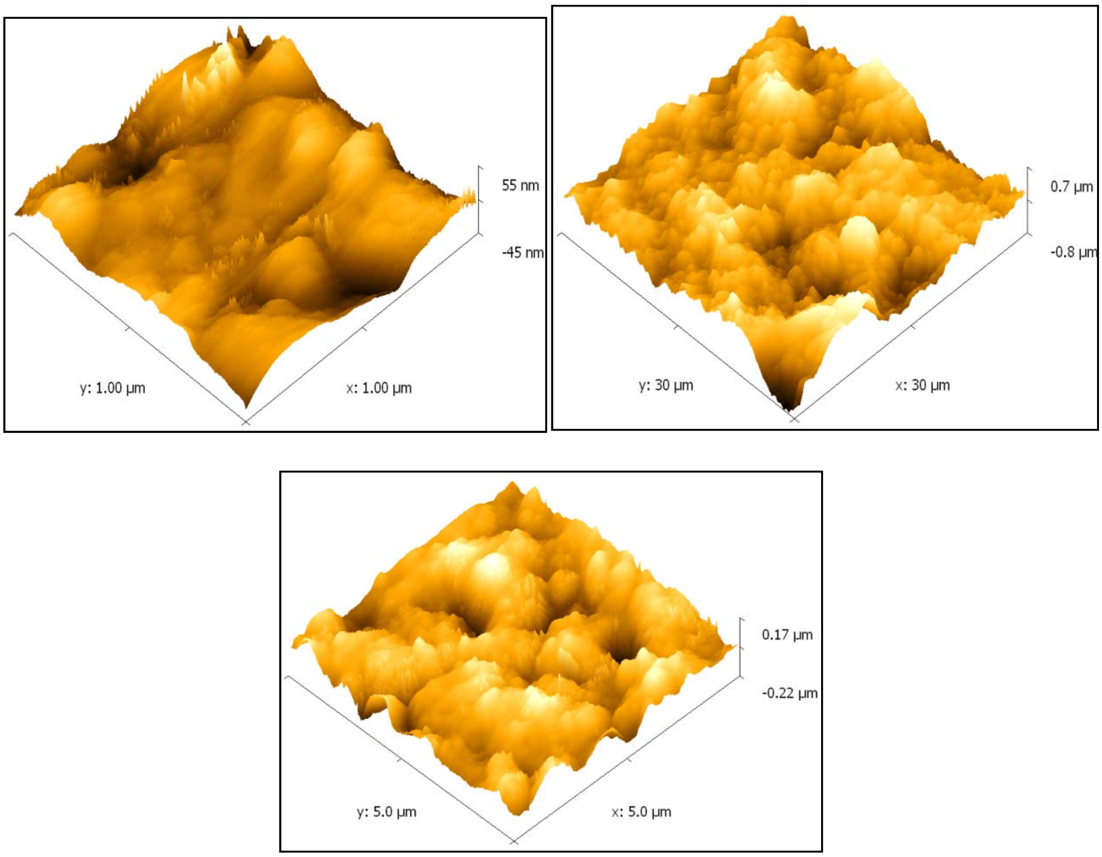

Fig.6. Atomic force microscopy images at used surface level for HERA-CERAM ceramic material

Unused area is almost smooth, roughness registering relatively small values (ranging between 0,03 nm - 13,8 nm) - figure 7

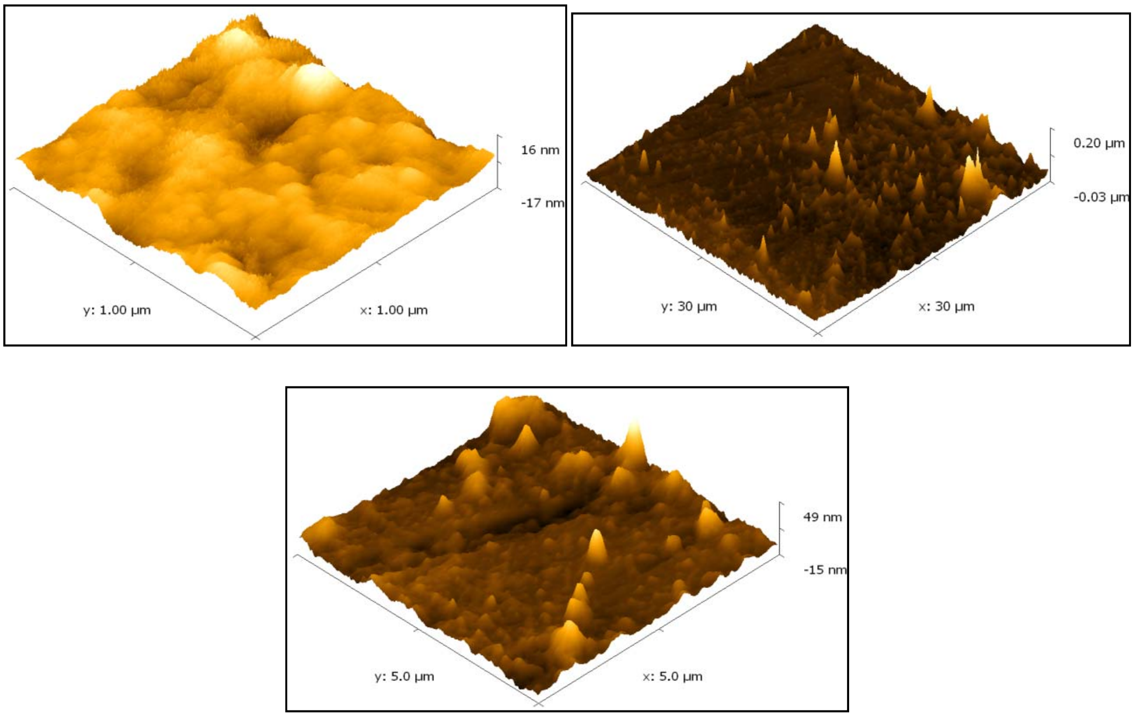

Fig. 7. Atomic force microscopy images at unused surface level for HERA-CERAM ceramic material

The values for used surface roughness compared with the unused surfaces of *HERA-CERAM* ceramic material are presented in table 3.

Table 3. Topographical characteristics of surfaces for HERA-CERAM material

	Topographical characteristics (nm)	Used surfaces	Unused surfaces
1 x 1	Average height	- 0,6 nm	- 0,03 nm
	Ra roughness	9,0 nm	2,40 nm
	RMS roughness	11,9 nm	3,26 nm
30 x 30	Average height	- 0,003 µm	0,5 nm
	Ra roughness	0,156 µm	9,2 nm
	RMS roughness	0,198 µm	13,8 nm
5 x 5	Average height	- 1,7 nm	0,05 nm
	Ra roughness	41,7 nm	3,54 nm
	RMS roughness	53,1 nm	5, 26 nm

Wear traces analysis shows that there are no significant roughness variations depending on loads for *VM13* material. In this case, it can see a reduction in the morphological changes of these layers, with a decrease in roughness due to increased Ni concentration in its composition. In figures 8 and 9 are highlighted changes in used surface quality compared to the unused area used for studied ceramic material.

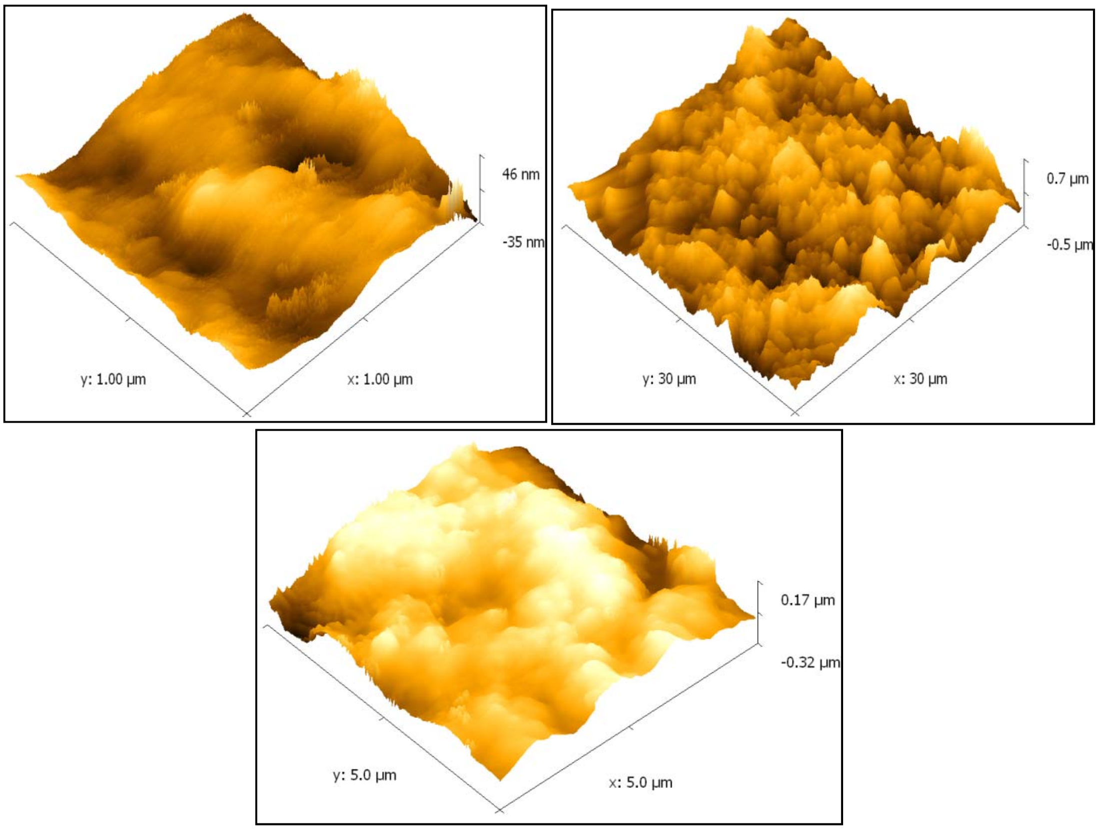

Fig. 8. Atomic force microscopy images at used surface level for VM13 ceramic material

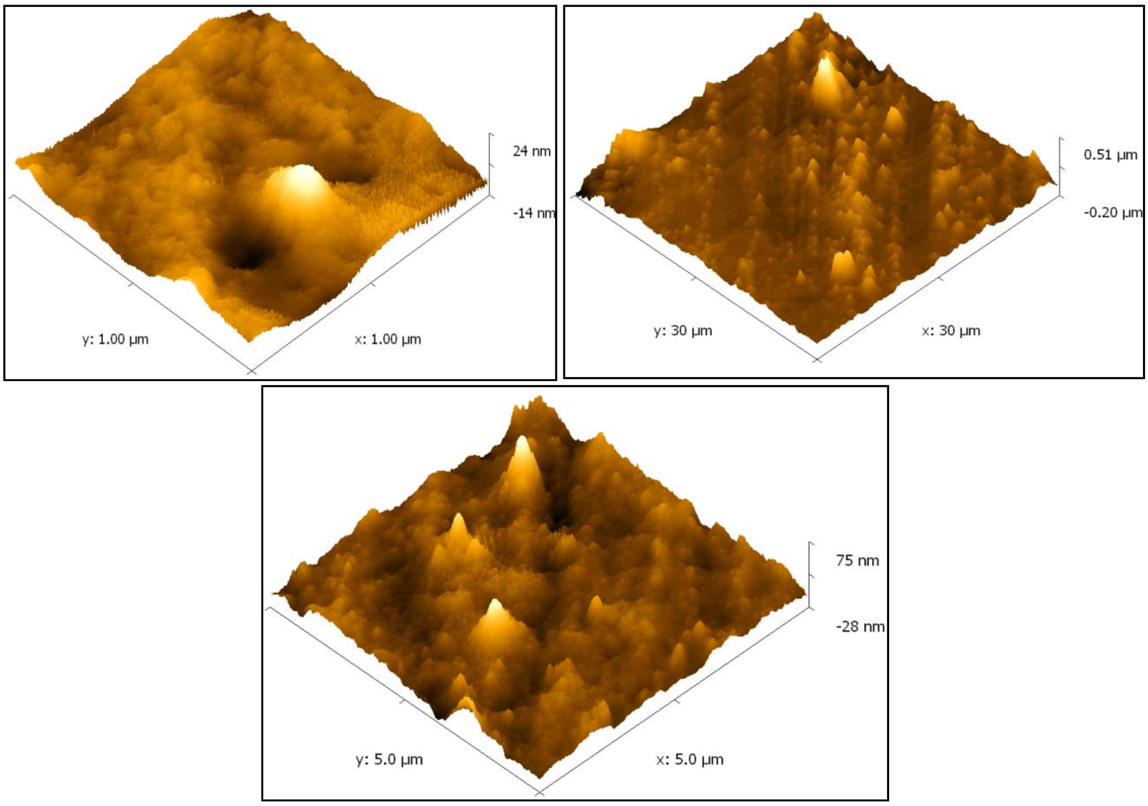

Fig. 9. Atomic force microscopy images at unused surface level for VM13 ceramic material

In table 4 are registered values registered by atomic force microscopy for used surface of *VM13* ceramic material compared to the unused surface.

Table 4. Topographical characteristics of surfaces for VM13 material

	Topographical characteristics (nm)	Used surfaces	Unused surfaces
1 x 1	Average height	0,03 nm	
	Ra roughness	8,05 nm	2,73 nm
	RMS roughness	9,84 nm	3,58 nm
30 x 30	Average height	0,001 µm	1,5 nm
	Ra roughness	0,125 µm	31,5 nm
	RMS roughness	0,159 µm	47,7 nm
5 x 5	Average height	- 0,5 nm	0,2 nm
	Ra roughness	65,7 nm	6,3 nm
	RMS roughness	81,9 nm	8,9 nm

It appears that roughness values for *VM13* ceramic material is part of a wide range, the highest value being 0,125 µm recorded when the sample material was driven by increased load for the same length of friction (which is in full agreement with the results previously obtained in tests carried out at this ceramic material), value would still be considered normal at oral level. The average height of roughness is closely related to the values obtained for Ra.

It is visible a lower roughness recorded at unused surface level; the range of values is of the order of nanometers.

As we mentioned at the beginning, another method for determining the surface quality of materials taken in this study, used Surtronic profilometry.

Ra roughness value measured at four types of materials based on profilometry ranges from 0,77 µm -to 2,47 µm.

Roughness diameter enlargement at the expense of depth, inadequate form curve profile associated with a roughness of 2,47 µm are characteristic aspects for *HERA-CERAM* material - figure 10.

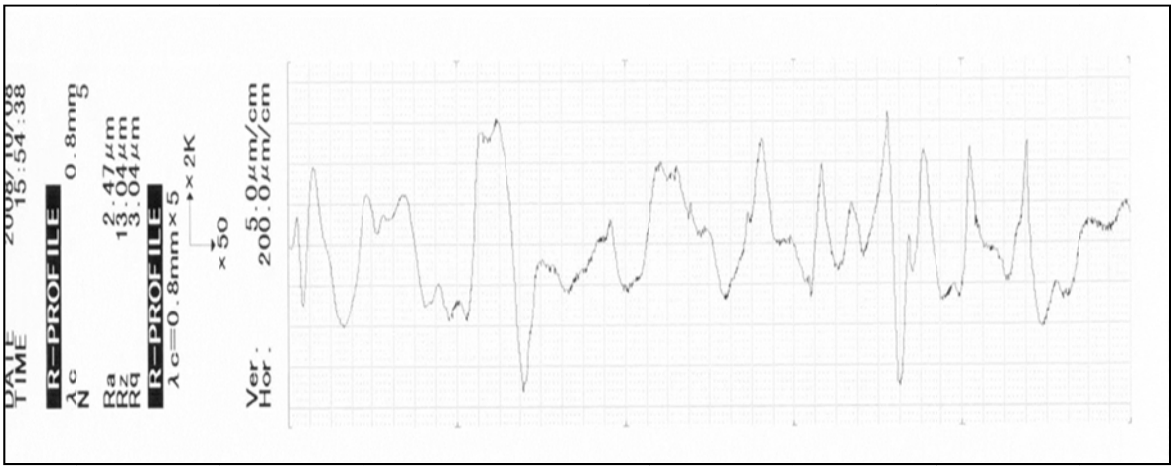

Fig. 10. Curve profile for HERA-CERAM surface.

Structural changes resulting from aging process affects the quality of the surface layer.

Aspects of profilometry for materials based on Si demonstrate a rounded profile roughness. If for *VM13* material - figure 11, roughness is is near the optimal value (Ra = 0,77 μm), although there is an obvious bump surface with wear facets, not the same it can be said about the other two materials, surface resulting from the aging process is quite rough, rough as it becomes more obvious as testing increases the diameter of the testing sphere – figs. 12 and 13.

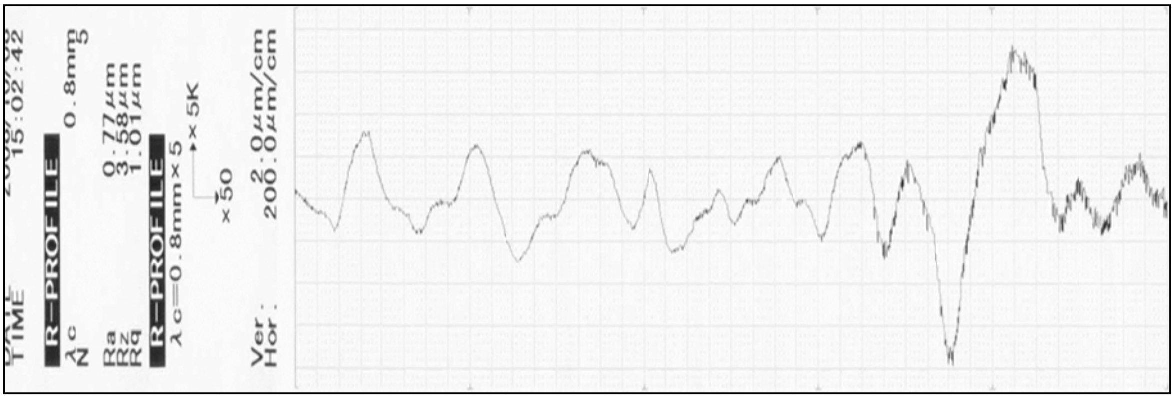

Fig. 11. Curve profile for VM13 surface

It appears the absence of significant differences concerning roughness of the two ceramic materials manufactured by IVOCLAR.

Profilometry analysis confirms that the aging process is accompanied by impaired surface quality material examined, with a roughness that promotes plaque buildup.

As the length of friction is increasing, in proportion to the amount of material used and increased surface roughness tested with obvious damage of resulting surface.

Variation of friction coefficient implies a variation of the surface roughness response.

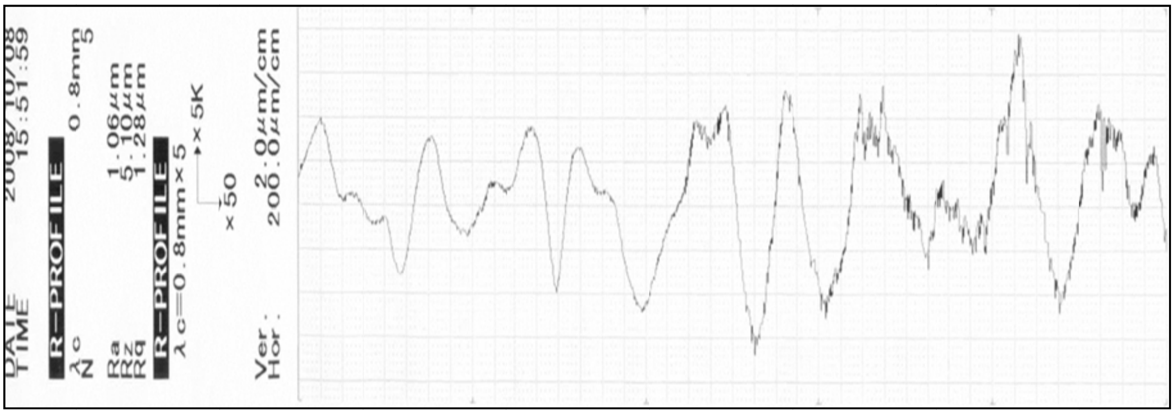

Fig. 12. Curve profile for D.SING surface

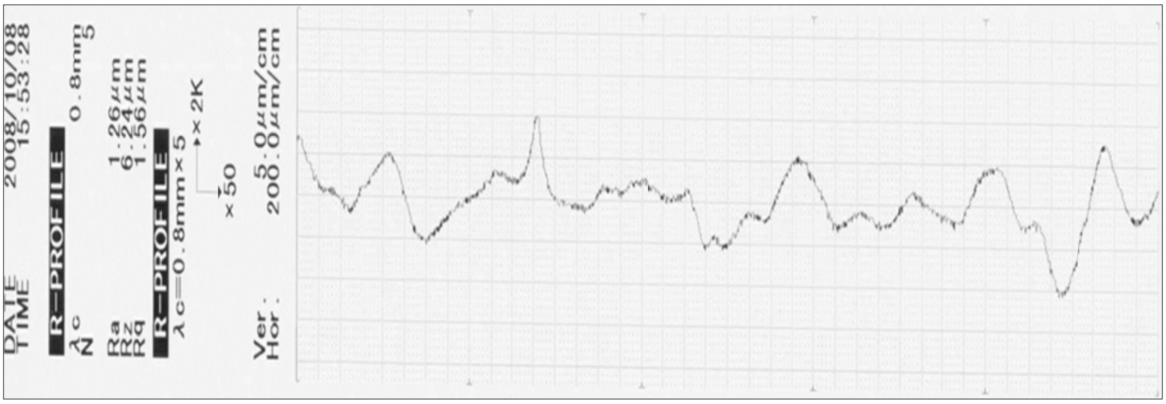

Fig. 13. Curve profile for IN LINE surface

In this study the profilometry measurements indicate variable values of roughness, but without reaching limiting values of plaque retention (0,2 μm), the roughness decreasing considerably as improving structural qualities. Material composition is a very important parameter and in most cases is critical to its behavior.

The measured profiles on the marked wear tracks of the surface of each material, confirms that the mark (patch of wear) can be approximated as a spherical segment (in full agreement with ball shape used for testing the wear resistance of abrasion). Roughness result therefore depends on the material, but also on the type of wear shape as test results.

The study notes a strong correlation between the amount of used material and the surface roughness; the roughness increases proportionally with it (for a volume of material transferred approximately 0,064353 mm^3 for *IN LINE*, and 0,061842 for *HERA-CERAM* ceramics, for example , is a roughness of about 1,26 μm or 2,47 μm). This is because the surface roughness of the material is the result of interaction of multiple factors associated with quantity (type, size, and distribution of particles), the type of matrix and effective link to the interface, all these parameters influencing roughness values.

It was thus confirmed that the wear factors are dependent on load for all roughness values of the four areas studied. For a constant force (in our case 0,35 N) acting on the material, it is found that there are wide variations of roughness in the first friction lengths, but significant increase in the length of friction is accompanied by significant increases in roughness. The wear coefficient increases with surface roughness resulting from the aging process.

A high rate of wear associated with an increased roughness may explain the increased amount of material used in the sliding movement. These residues are characteristic for wear phenomenon of three bodies, phenomenon frequently encountered at oral level, which could lead to an initial decrease of studied surface roughness.

Although there is a close relationship between wear rate and alleged roughness, the only one phenomenon that occurs is represented by an increased roughness which implies the transition in wear process. The wear coefficient variation is closely related to tribo-chimical reactions occurring between the ceramic material, the metal support and lubricant; this unexpected phenomenon can only occur if the surface is very rough. These tribo-chemical reactions are dependent on oral pH and can be accelerated in the oral cavity of factors related to the chemical reactivity of the patient.

VM13 ceramic material showed a significantly lower roughness compared to other types of ceramics. The observed differences in the roughness profile of the studied materials could be attributed to micro-structural characteristics of each type of ceramic or size and shape of cristals.

As micro-structure, *VM13* ceramics shows a more homogeneous distribution in the glass phase, which causes smooth surfaces as it was obtained in our study.

Irregular shape of the crystals of the crystalline phase causes increased roughness of CERAM HERA-CERAM facets.

Although crystalline content is low, regular polygonal shape of crystals of ceramics structure IN LINE and D.SING leads to an increased roughness.

Increased roughness of ceramics materials causes appearance of used materials and the stress distribution can be correlated with parameters Ra, Rz, Rmax.

The stress distribution and the resistance values can be directly correlated with roughness parameters Ra, Rz, Rmax.,for all studied ceramics. Unused ordinary ceramics present a low mechanical stability. If the needle tip of profilometer is in direct contact with roughness then will appear changes of the defect size of the surface and the roughness may affect the fatigue strength, clearly evidenced by the results of our study (similar results were found in studies presented in the literature) [1].

D.SING is a matrix of reinforced glass with crystalline particles. *HERA-CERAM* has a polycrystalline structure containing particles with stabilizing role. The fine microstructure provides a low roughness for *VM13* material. However, micro-structural differences are reflected in the value of resistance value under a maximum stress and are similar with other literature studies [2, 3, 4, 5].

There have been observed a number of differences between the two test methods. These differences are caused by variation in the length of friction occurred, length on which the determination was made or due to the device technical qualities. Due to mechanical displacement of the profilometer sensor may occur several successive errors, the needle draggings on hemispheric shape of the used surface leading to increased roughness values as is confirmed by our research results. When the diamond needle able to scan the sample samples moves on a flat wear facets of surface, a sliding motion occurs over the edge facets in a sharp angle and slope, resulting in increased values. Other possible sources of errors are: distortion of the scanning needle, needle geometric shape that can create problems especially when there are scanning some steeper walls by 45^0.

The sensor cannot detect narrow grooves at the rough area level and the information about localized peaks in depth areas are interspersed during scanning and a loss of information can occur concerning the deep points of facets; these deficiencies are solved through atomic force microscopy. This may explain why the sensor could measure smaller vertical loss.

However, the wear facets include few abrupt angles, low uneven surface or reduced vertical losses, all of which may be as a result the affecting of accuracy determinations.

In general, ceramics used in complex oral rehabilitation presents an increased crystalline content, so that the molded ceramic surface on the metalic support should be as smooth, in order to minimize bacterial colonization and bacterial biofilm formation.

In oral environment, the increasing of ceramic surface roughness can lead to increased accumulation of bacterial biofilm at the level of restorative material.

Initially, microorganisms adhesion start with the surface irregularities of ceramic material used in restoration and gradually spreads to the internal surface. Thus, the material surface roughness influences both bacterial adhesion and rapidly maturation of formed biofilm, which has a number of great clinical implications.

A large roughness is responsible for a substantial loss of aesthetic restoration. Temperature changes in the oral environment would result in a torsional stress in restorative ceramic mass as results of differences in thermal expansion coefficients of the two materials in contact.

If ceramic restorations are rugged, by abrasion or fracture processes, it is possible to be installed at oral level the three bodies wear process with an significant acceleration of this process.

Finishing procedures may affect the integrity of ceramic restoration, causing increased surface roughness, which is accompanied by gingival inflammation and effect reactions to surrounding of soft tissue. In addition, occlusal adjustments can cause excessive wear of the antagonist tooth with changing distribution of occlusal forces to the level of restoration itself.

In addition, the increased of surface roughness restoration may influence aging period of opponent enamel; the reply to this wear or other restorative material in the presence of a rough surface, but with a hardness similar to that of ceramic, not significantly different because the fundamental mechanism of material movement during abrasion wear is the same [6, 7, 8].

4. Conclusions

Following the results of this study can be said that:

• the increased volume of used material is associated with the increased profile of studied ceramic surface roughness, although the wear factors remain independent of load;

• wear process is accompanied by the appearance of deformation and fractures in contact with asperities, a phenomenon that is followed by the residual layer interfacial deformation;

• there is a difference, sometimes striking, between the values obtained using the two possibilities of determination (atomic force microscopy and profilometry), differences which were influenced both by the accuracy of measurements and of the length of friction, and the technical equipment performances;

• at oral level, the clinical implications of an increased roughness are multiple: restorations become rough due to changes during the performance of stomatognathic system; it is increased the wear process already installed, while causing an increased rate of damage on the antagonist arch.

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