

Application of atomic force microscopy in investigations of dental alloy surfaces

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SUMMARY

Introduction The aim of this informative paper is to show the importance and key role of atomic force microscopy, on one hand, in quantitative determination of the physicochemical changes on the surfaces of dental materials due to their exposure to acids and, on the other hand, in quantification of changes in the physicochemical properties of the surfaces of dental alloys after their processing and the consequences of the processing itself on the quality and applicability of the same.

Methods Atomic force microscopy was used to obtain data quantitatively describing nanoscale changes in Co-Cr dental alloys caused by exposure to formic acid over a 7-day period and to describe the effects of electropolishing and black brush polishing on the surface roughness of dental alloys.

Results Analysis of the topography and roughness of the surfaces of alloys treated with a black brush and electropolishing shows that both methods are applicable in dentistry and leave defects of insufficient size for microorganisms. Treatment of the studied alloys with formic acid leads to an increase in surface roughness, indicating the presence of corrosion processes, especially in the areas of interdendritic nanocrystals.

Conclusion The aforementioned effects of processing and treatment of Co-Cr alloys can be followed in great detail using the technique of atomic force microscopy, especially by analysing the surface topography and its roughness. The method is of extraordinary importance for the evaluation of the use of Co-Cr alloys in dentistry.

Keywords: atomic force microscopy; surface roughness; Co-Cr dental alloys; formic acid; electropolishing

INTRODUCTION

Co-Cr alloys are materials that have been used for long time as dental materials due to their exceptional mechanical properties, especially their strength [1]. Without going into the description of the structure of the alloys themselves, one of their essential properties is their surface roughness [2]. In order to be used as dental materials, Co-Cr alloys require processing, which includes their polishing [3, 4]. In addition, the surface of dental alloys is extremely important for their application, because the surface roughness depends on the interaction of the surface with oral fluid, formation of dental plaque, and interaction with microorganisms and fluids in the oral cavity. As a result of interaction with oral fluids, chemical reactions and corrosion occur, which can alter the physical and chemical properties of dental alloy surfaces and thus limit their use.

Atomic force microscopy is one of the techniques that can be used to determine topography of surfaces at the nanoscale. Therefore, it is of crucial importance in assessing the quality of dental materials, especially their surfaces [5]. Without going into the description of the basic working principle of the technique itself, an important parameter determined by this method, in addition to topography, is the surface roughness parameter, which indicates the size of surface defects suitable for microorganism uptake [6].

In this informative paper, the approach to the problems of determining the topography and roughness of

the previously studied surfaces of Co-Cr alloys and their changes that occurred after the processing of the alloys themselves by electropolishing and polishing with a black brush is described, focusing on the use of atomic force microscopy as a key technique for obtaining arguments important for the application of the alloy in dentistry [4]. In addition, a detailed analysis of the topography of Co-Cr alloy surfaces before and after corrosion induced by formic acid treatment over a 7-day period is presented and explained.

THE EFFECT OF POLISHING ON THE TOPOGRAPHY AND SURFACE ROUGHNESS OF CO-CR ALLOYS

The preparation and processing of the Co-Cr alloys themselves have been described in previous studies [3, 4]. Briefly, after casting, the alloy samples were cleaned in a vacuum casting machine at 1420°C, sandblasted, and then polished with a black brush and electropolishing. After that, they were additionally polished with a rotating brush for 15 minutes. The Co-Cr alloys were taped to a metal disk and imaged in contact mode (Nanoscope III with control program, Bruker Instruments, Santa Barbara, USA). Imaging was performed with a Bruker NP probe whose nominal probe constant is $k_{\text{nom}} = 0.32 \text{ N/m}$. The location of interest for surface imaging is selected with an optical camera. The topographies of dental alloy surfaces

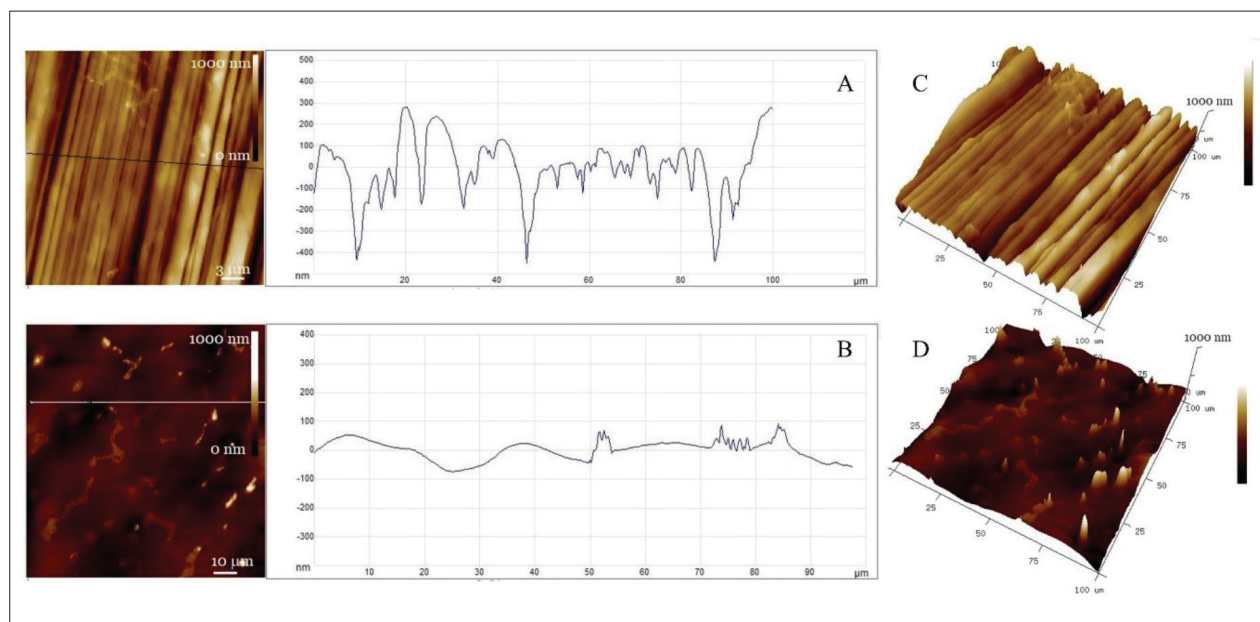


Figure 1. 2D topographic view of the heights and cross-sectional profile along the marked line of the $30 \times 30 \mu\text{m}^2$ Co-Cr alloy surface treated with a black brush (A) and electropolishing (B). 3D view of the heights of the Co-Cr alloy treated with a black brush (C) and electropolishing (D). The vertical scale is 1000 nm.

Slika 1. 2D topografski prikaz visine i profila poprečnog preseka duž označene linije legure Co-Cr površine $30 \times 30 \mu\text{m}^2$ obradene rotirajućom četkom (A) i elektropoliranjem (B). 3D prikaz visina legure Co-Cr obradene crnom četkom (C) i elektropoliranjem (D). Vertikalna skala je 1000 nm.

polished with black brush and electropolishing, their cross-sectional profiles, and 3D topographic views of heights are shown in Figure 1.

As can be seen in Figures 1C and 1D, the three-dimensional topographic images of the heights corresponding to the two different polishing methods are completely different. After polishing, the fine dendritic wave structure and the resulting interdendritic nanocrystals on the surface of the Co-Cr alloy are damaged by newly formed dominant scratches distributed at different spacing and scratch depths, reaching values of up to 400 nm. In contrast to the surface treated with a black brush, the surface of Co-Cr alloy that has been electropolished is dominated by dendrites with relatively smoothed wavy surfaces and interdendritic areas with clusters of nanocrystals emerging from the smoothed surface with an average height of 100-200 nm. For the application of polished Co-Cr alloys, it is important that the surface in direct contact with liquids is as smooth and flat as possible, with the lowest values of roughness parameters. For this reason, an analysis of the cross-section profile of the two polished Co-Cr alloys was performed, as shown in Figures 1A and 1B. This type of analysis must be performed on multiple surfaces to determine the mean and standard deviation. To gain insight into the smoothness of alloy surfaces with a higher degree than local along the section line, roughness analysis was performed on larger alloy surfaces ($30 \times 30 \mu\text{m}^2$ and $100 \times 100 \mu\text{m}^2$) in addition to the cross-section profile, and the values of the roughness parameters of one analysis are listed in Table 1. It is common to consider R_a (roughness parameter), R_q (root mean square of roughness) and Z range (the largest vertical distance between the highest and lowest measured position on the surface).

Table 1. Roughness parameters for Co-Cr dental alloys treated with a black brush and by electropolishing

Tabela 1. Vrednosti parametara hrapavosti za dentalne legure Co-Cr pre i posle tretmana mravljom kiselinom

Polishing treatment Tretman poliranjem	Analyzed surface area / μm^2 Analizirana površina / μm^2	aR_a /nm	bR_q /nm	cZ range/nm
Black brush Crna	30×30	84	112	684
	100×100	115	154	1236
Electropolishing Elektropoliranje	30×30	22	33	358
	100×100	44	60	877

aR_a – average roughness; bR_q – the root mean square of the roughness; cZ range – the greatest vertical distance between the highest and the lowest measured position on the surface

aR_a – srednja hrapavost; bR_q – koren srednjeg kvadrata hrapavosti; cZ raspon

– najveća vertikalna udaljenost između najviše i najniže izmerene pozicije na površini

It should be noted that roughness values increase with increasing size of the surface under investigation. This is especially important when analyzing inhomogeneous surfaces. Therefore, it is always better to analyze as large a surface area of the alloys as possible to get a realistic view of the topography and roughness of the alloys. Since the values of the structures emerging from the wavy surface and the depth of the scratches are smaller than the dimensions of microorganisms (1–4 μm) in the oral cavity, the aforementioned polishing effects are not representing an obstacle to the use of both treated alloys in dentistry.

Finally, the procedure for analyzing surfaces of Co-Cr-treated alloys is presented, which is necessary to verify whether the surface properties of the alloy are satisfactory.

In conclusion, the method and technique necessary and sufficient to evaluate the validity of processing alloys by various methods for their safe use in dentistry is described here.

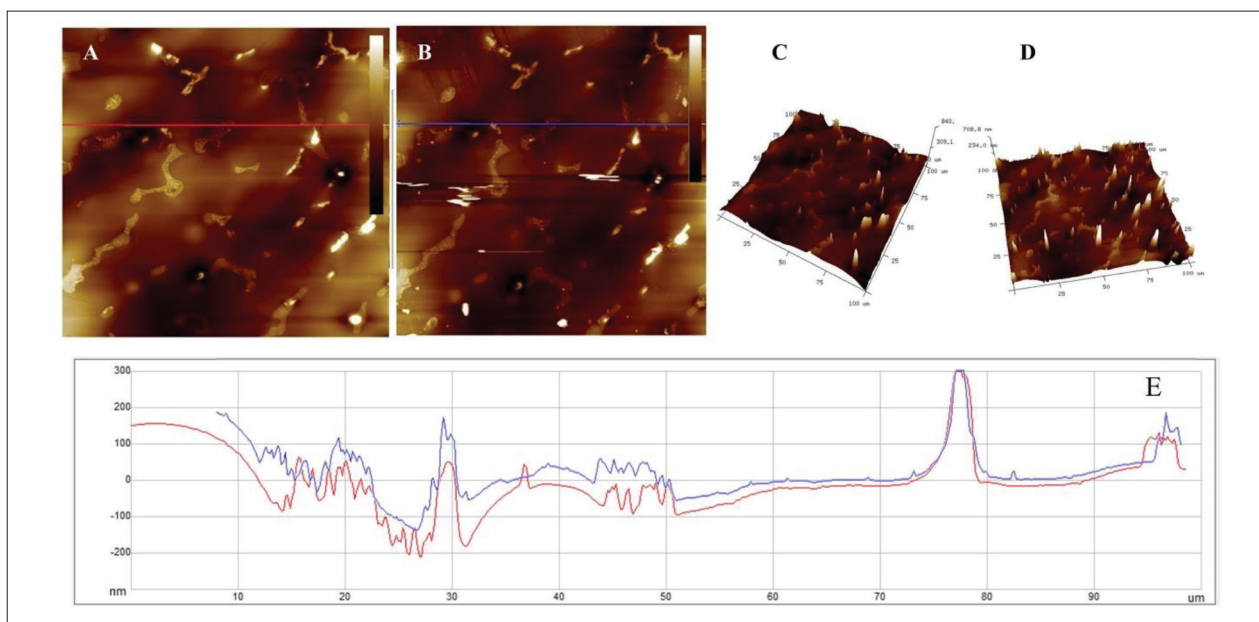


Figure 2. 2D and 3D topographic representation of the heights of Co-Cr alloy with a surface area of $100 \times 100 \mu\text{m}^2$ before (A and C) and after 7-day treatment with formic acid (B and D), and cross-sectional profile along the marked lines (E) before (red) and after (blue).
Slika 2. 2D i 3D topografski prikaz visina legure Co-Cr površine $100 \times 100 \mu\text{m}^2$ pre (A i C) i posle sedmodnevnog tretmana mravljom kiselinom (B i D), kao i profil poprečnog preseka duž označenih linija (E) pre (crveno) i posle (plavo). Vertikalna skala je 1000 nm.

EFFECT OF CORROSION ON TOPOGRAPHY AND SURFACE ROUGHNESS OF CO-CR ALLOYS

Having described the effect of polishing on topography and surface roughness, a very important information that atomic force microscopy can and does provide is the change in the surface due to corrosion caused by treatment with formic acid. It is critical that identical areas are imaged before and after acid treatment. Indeed, whenever alloy surfaces are treated, they must be removed from the unit, the treatment itself performed, and the alloys returned for imaging. Therefore, it is necessary to develop a method that ensures the analysis of completely identical locations on the alloy surfaces, which was the case in this study.

Briefly, the alloy samples were imaged at four different locations before treatment, and the size of the imaged area varied ($100 \times 100 \mu\text{m}^2$; $30 \times 30 \mu\text{m}^2$; and $10 \times 10 \mu\text{m}^2$). Figure 2 (A and B) and Figure 2 (C and D) show an example of 2-D and 3-D topographic representation of the surface heights of Co-Cr alloys before and after 7-day treatment with formic acid.

Topographic images of the Co-Cr alloy surface show typical dendritic structures with relatively smooth, undulating surfaces and interdendritic regions with clusters of nanocrystals.

The apparently identical 2D and 3D topographic surface height images of Co-Cr alloys before and after formic acid treatment, shown in Figure 2(A) and (B) and 2(C) and (D), are examined in more detail by analyzing the profile of the cross-section shown in Figure 2(E). The cross-sectional profile of the surface of the Co-Cr alloy before treatment is shown in red, while the cross-sectional profile after the 7-day treatment with formic acid is shown in blue. It is immediately noticeable that the effect of formic acid affects the interdendritic regions of

the Co-Cr alloy surface the most. The height of the nanocrystals emerging from the smoothed surfaces averages between 100 and 200 nm before and after treatment. However, what is immediately noticeable is that the contours of the nanocrystals are rounded before treatment, while they are lower, sharper, and rougher after treatment, suggesting that a corrosion process is involved. Detailed analysis showed that about 2.5% of the nanocrystals were corroded at their height. In this way, it is very practical for the practical application of the cross-section method in analyzing the corrosion of dental materials. However, in order to obtain a general picture of the surface processes, it is necessary to analyze the roughness of the surfaces of Co-Cr alloys before and after treatment with formic acid. The results presented in Table 2 for a selected surface ($100 \times 100 \mu\text{m}^2$) show that the roughness of the Co-Cr surface increased by 9% in R_a , 24% in R_q , and 142% in Z, indicating that the corrosion process after treatment with formic acid is significant. The results of extensive investigations are reported in the literature [4].

Table 2. Roughness parameter values for Co-Cr dental alloys before and after treatment with formic acid

Tabela 2. Vrednosti parametara hrapavosti za dentalne legure Co-Cr pre i posle tretmana mravljom kiselinom

Treatment Tretman	Analyzed surface area / μm^2 Analizirana površina / μm^2	R_a /nm	R_q /nm	Z range/nm
-	100×100	63.4	81.7	1028
7 days 7 dana	100×100	69.1	101	2487

CONCLUSION

Atomic force microscopy is a very powerful method for the analysis of all dental materials, especially Co-Cr dental

alloys. It allows imaging of various surfaces and visualization of the topography of surfaces in two and three dimensions. Moreover, it allows not only a detailed insight into the physicochemical properties of surfaces, especially surface roughness, but also the monitoring of processes taking place on the surfaces themselves at the nanoscale.

This paper presents the application of atomic force microscopy to the research already conducted on dental materials, which is not only useful but also necessary for the application of dental materials in dentistry. Based on the section profile of the examined surface along a line, insight into local surface properties such as roughness is obtained, while roughness analysis is applicable to larger surfaces and is a useful parameter for monitoring the quality of the dental materials themselves.

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Primena mikroskopije atomskih sila u istraživanjima površina dentalnih legura

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KRATAK SADRŽAJ

Uvod Cilj ovog informativnog rada je da prikaže važnost i ključnu ulogu mikroskopije atomskih sila, s jedne strane, u kvantitativnom određivanju fizičko-hemijskih promena na površinama dentalnih materijala usled njihove izloženosti kiselinama, a s druge strane u kvantifikovanju promena fizičko-hemijskih svojstava površina dentalnih legura posle njihove obrade, te posledicama same obrade na njihov kvalitet i primenljivost.

Metode Mikroskopijom atomskih sila dobijeni su podaci koji kvantitativno opisuju promene na nanoskali na dentalnim legurama Co-Cr nastale usled izloženosti delovanju mravlje kiseline kroz period od sedam dana, a opisani su i efekti elektropoliranja i poliranja crnom rotirajućom četkom na hrapavost površina.

Rezultati Analiza topografije i hrapavosti površina legura obrađenih rotirajućom crnom četkom nakon elektropoliranja ili samo elektropoliranjem pokazuje da su obe metode primenjive u stomatologiji ostavljajući defekte dimenzija nedostatnih za adherenciju mikroorganizama. Izlaganje analiziranih legura mravljoj kiselini dovodi do povećanja hrapavosti površina ukazujući na prisutnost procesa korozije, naročito na područjima interdentskih nanokristala.

Zaključak Navedeni efekti obrade i tretmana legura Co-Cr vrlo se detaljno mogu pratiti tehnikom mikroskopije atomskih sila, specifično analizom topografije površina i analizom njene hrapavosti. Metoda pokazuje izuzetnu važnost za procenu primene legura Co-Cr, a takođe i drugih legura u dentalnoj medicini.

Ključne reči: mikroskopija atomskih sila; hrapavost površina; dentalne legure Co-Cr; mravlja kiselina; elektropoliranje

UVOD

Legure Co-Cr su materijali koji se zbog svojih izuzetnih mehaničkih svojstava, naročito zbog svoje čvrstoće, već dugo koriste kao stomatološki materijali [1]. Ne ulazeći u opis strukture samih legura, jedno od njihovih bitnih svojstava jeste njihova površinska hrapavost [2]. Da bi se mogle upotrebiti kao stomatološki materijal, legure Co-Cr moraju se obraditi, što uključuje i njihovo poliranje [3, 4]. Nadalje, sama površina dentalnih legura od izuzetne je važnosti za njihovu primenu jer o hrapavosti njihove površine zavisi interakcija površine s oralnim tečnostima, formiranje dentalnog plaka, te interakcija s mikroorganizmima i tečnostima prisutnim u oralnoj šupljini. Usled interakcije s oralnim tečnostima dolazi do hemijskih reakcija i korozije koje mogu promeniti fizičko-hemijska svojstva površina dentalnih legura i na taj način ograničiti njihovu primenu.

Mikroskopija atomskih sila jedna je od tehnika kojom se može odrediti topografija površina na nanoskali. Stoga je njena uloga od ključne važnosti u proceni kvaliteta dentalnih materijala, posebno njihovih površina [5]. Bez ulaženja u opis osnovnog principa rada same tehnike, osim topografije, važan parametar koji se određuje ovom metodom jeste parametar hrapavosti površine, koji ukazuje na veličinu površinskih defekata pogodnih za smeštaj mikroorganizama [6].

U ovom informativnom radu opisać se pristup problemima određivanja topografije i hrapavosti već istraživanih površina legura Co-Cr i njihovih promena nastalih posle obrade samih legura elektropoliranjem i poliranjem rotirajućom crnom četkom s naglaskom na primeni mikroskopije atomskih sila kao ključne tehnike u dobijanju argumenata važnih za primenu legura u stomatologiji [4]. Nadalje, biće prikazana i objašnjena detaljna analiza topografije površina legura Co-Cr pre i posle korozije inducirane uranjanjem u mravlju kiselinu u periodu od sedam dana.

EFEKAT POLIRANJA NA TOPOGRAFIJU I HRAPAVOST PVRŠINA LEGURA CO-CR

Postupak pripreme i obrade samih legura Co-Cr opisan je u ranijim studijama. Ukratko, posle izlivanja pomoću mašine za vakuumsko livenje pri 1420° C, uzorci legure su očišćeni od uložne mase i posle toga peskareni i zatim elektropolirani. Nakon toga su dodatno polirani rotirajućom četkom u trajanju od 15 minuta. Legure Co-Cr su zalepljene lepljivom trakom za metalni disk te oslikane u kontaktnom načinu rada AFMa (nanoskopom III sa kontrolnim programom, Bruker Instruments, Santa Barbara, SAD), a snimanje je izvedeno sondom Bruker NP, čija je nominalna konstanta probe $k_{nom} = 0,32 \text{ N/m}$. Lokacija od interesa za oslikavanje površina odabrana je optičkom kamerom. Topografije površina dentalnih legura elektropolirane su i polirane crnom rotirajućom četkom i njihovi profili poprečnog preseka i 3D topografski prikazi visina prikazani su na Slici 1.

Kao što se vidi iz slika 1C i 1D, trodimenzionalna topografska slika visina kojima odgovaraju dve različite metode poliranja potpuno je različita. Fina dentska valovita struktura i izranjajući interdentski nanokristali na površini legure Co-Cr posle elektropoliranja narušeni su novoformiranim dominantnim brazdama međusobno raspoređenim na različitim udaljenostima, te dubinama brazda koje dosežu vrednosti i do 400 nm. Za razliku od površine obrađene rotirajućom četkom, površinom legure Co-Cr koja je podvrgnuta samo elektropoliranju dominiraju dendriti s relativno zaglađenim valovitim površinama i interdentskim područjima s nakupinama nanokristala koja izranjaju iz zaglađene površine s visinama u preseku do 100 do 200 nm. Za primenu poliranih legura Co-Cr važno je da je površina koja dolazi direktno u kontakt s tečnostima što glađa i ravnija, sa što manjim vrednostima parametara hrapavosti. Zato je analiziran profil poprečnog preseka obe polirane legure Co-Cr, koji je prikazan na slikama 1A i 1B. Ovakvu analizu potrebno je uraditi na više površina i dobiti srednju vrednost i standardno odstupanje. Da bi se dobio uvid u

glatkoću površina legura višeg stupnja od lokalnog uzduž linije preseka, osim profila poprečnog preseka, analizirana je hrapavost na većim površinama legure ($30 \times 30 \mu\text{m}^2$ i $100 \times 100 \mu\text{m}^2$), a vrednosti parametara hrapavosti jedne analize prikazane su u Tabeli 1. Uobičajeno je da se razmatraju parametri R_a (parametar hrapavosti), R_q (koren srednjeg kvadrata hrapavosti) i Z raspon (najveća vertikalna udaljenost između najviše i najniže izmerene pozicije na površini).

Ovde treba imati na umu da vrednost hrapavosti površina raste s povećanjem analizirane površine. To je naročito važno kad se analiziraju površine koje nisu homogene. Zato je uvek bolje analizirati maksimalno moguće veliku površinu legura kako bi se dobio realan uvid u topografiju i hrapavost legura.

Budući da su vrednosti struktura koje izranjaju iz valovite površine i dubina brazda manje od dimenzija mikroorganizma ($1-4 \mu\text{m}$) u oralnoj šupljini, navedeni efekti poliranja ne predstavljaju prepreku za upotrebu obe metode u obradi legura u stomatologiji.

Zaključno, prikazan je postupak analize površina obrađenih legura Co-Cr koji je nužan za ispitivanje da li su površinska svojstva legure zadovoljavajuća.

Zaključno, ovde su opisani način i tehnika koji su nužni i dovoljni za procenu valjanosti obrade legura različitim metodama za njihovu sigurnu primenu u stomatologiji.

EFEKAT KOROZIJE NA TOPOGRAFIJU I HRAPAVOST POVRŠINA LEGURA CO-CR

Posle opisanog efekta poliranja na topografiju i hrapavost površina, vrlo važnu informaciju koju daje mikroskopija atomskih sila jeste njihova promena usled korozije inducirane tretmanom kiselinama, mravljom i octenom. Pritom je od ključne važnosti da se oslikaju identična područja od interesa pre i posle tretmana kiselinama. Naime, bilo koji tretman površina legura zahteva njihovo uklanjanje s uređaja, sam tretman i ponovno vraćanje legura za oslikavanje. Zato je potrebno razviti metodologiju kojom bi se osigurala analiza potpuno identičnih lokacija na površinama legura, što je u ovom istraživanju i bio slučaj.

Ukratko, uzorci legura pre tretmana oslikani su na četiri različite pozicije, dok je veličina oslikanog područja varirala ($100 \times 100 \mu\text{m}^2$, $30 \times 30 \mu\text{m}^2$ i $10 \times 10 \mu\text{m}^2$). Primer topografskog 2D i 3D prikaza visine površine legura Co-Cr pre i posle sedmodnevnog tretmana mravljom kiselinom prikazan je na Slici 2 (A i B) i 2 (C i D).

Topografske slike površine legure Co-Cr pokazuju tipične dendritske strukture s relativno zaglađenim valovitim

površinama te interdendritskim područjima s nakupinama nanokristala.

Naizgled identične topografske 2D i 3D slike visina površina legura Co-Cr pre i posle tretmana mravljom kiselinom prikazanih na Slici 2 (A) i (B) i 2(C) i ((D) proučavaju se detaljno analizom njihovog profila poprečnog preseka prikazanog na Slici 2(E). Na slici je crvenom bojom naznačen profil poprečnog preseka površine legure Co-Cr pre tretmana, dok je plavom bojom naznačen profil poprečnog preseka posle sedmodnevnog tretmana mravljom kiselinom. Odmah se uočava da je delovanje mravlje kiseline najviše uticalo na interdendritska područja površine legure Co-Cr. Visina nanokristala koja izranjaju iz zaglađenih površina kreću se u proseku od 100 do 200 nm pre i posle tretmana. Međutim, odmah se uočava da su obrisi nanokristala pre tretmana zaobljeni, dok su posle tretmana niži, zaoštreni i hrapavi sugerišući da se radi o procesu korozije. Detaljnom analizom se ustanovilo da je korodiralo oko 2,5% nanokristala s obzirom na visinu. Na ovaj način je vrlo praktično pokazana primena metode poprečnog preseka u analizi korozije dentalnih materijala. No, želeći da se dobije generalna slika površinskih procesa, nužno je i ovde analizirati hrapavosti površina legura Co-Cr pre i posle tretmana s mravljom kiselinom. Rezultati prikazani u Tabeli 2 na jednoj odabranoj površini ($100 \times 100 \mu\text{m}^2$) pokazuju da se hrapavost površine Co-Cr povećala u R_a za 9%, R_q za 24% te Z rasponu za 142%, što ukazuje na to da je posle tretmana mravljom kiselinom proces korozije značajan. Rezultati opsežnog istraživanja navedeni su u literaturi [4].

ZAKLJUČAK

Mikroskopija atomskih sila vrlo je snažna metoda za analizu svih dentalnih materijala, a specifično i dentalnih legura Co-Cr. Ona omogućuje oslikavanje različitih površina i vizualizaciju topografije površina u dve i tri dimenzije. Osim navedenog, ona omogućuje ne samo detaljan uvid u fizičko-hemijska svojstva površina, posebno hrapavost površina, nego i praćenje procesa koji se događaju na samim površinama na nanoskali.

Ovaj rad predstavlja primenu mikroskopije atomskih sila na već sprovedenim istraživanjima stomatoloških materijala, a koja su ne samo korisna nego i neophodna za primenu stomatoloških materijala u stomatologiji. Pomoću profila preseka analizirane površine duž jedne linije dobija se uvid u lokalna svojstva površina kao što su hrapavost, dok je analiza hrapavosti primenjiva na većim površinama i koristan je parametar praćenja kvaliteta samih dentalnih materijala.