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Utjecaj obrade erozimatom na mikrostrukturu površine titanija dobivenu metalurgijom praha

Effects of Wire EDM on the Microstructure of P/M Titanium Samples

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Sažetak

Komercijalno čisti titanij (CP Ti) se koristi u dentalnoj medicini zbog biokompatibilnosti, dobrih mehaničkih svojstava i otpornosti na koroziju. Konvencionalni proizvodni procesi izrade takvog titanija mogu utjecati na kvalitetu površine uzoraka i rezultirati slabim vezanjem CP Ti s dentalnom keramikom. Zato se uvode nove tehnologije proizvodnje titanija, primjerice metalurgija praha i oblikovanje na erozimatu s žicom (WEDM). Svrha ovog istraživanja jest odrediti utjecaj WEDM-a na površinu uzoraka P/M CP Ti proizvedenih za ispitivanje vezne čvrstoće prema normi ISO 9693. **Materijali i metode:** Osam uzoraka P/M CP Ti dimenzija prema normi ISO 9693 proizvedeno je korištenjem WEDM-a i podijeljeno u dvije grupe – u neobrađene i brušene. Površine obje grupe uzoraka analizirane su metodama SEM, EDS i XDR. **Rezultati:** Analize neobrađenih uzoraka metodama SEM i EDS pokazuju tanki sloj različitog sastava i frakture. Brušeni uzorci imaju homogenu strukturu bez fraktura. Analiza metodom XDR pokazuje visoku koncentraciju titanijevih oksida na površini neobrađenih uzoraka, a nakon brušenja dobivena je samo čista α-faza. **Zaključak:** WEDM je metoda prikladna za proizvodnju uzoraka prema normi ISO 9693, ako se uzorci naknadno bruse.

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Ključne riječi
titanij; erozimati sa žicom; vezna čvrstoća; metalurgija praha; svojstva površina

Uvod

Komercijalno čisti titanij (CP Ti) se koristi u dentalnoj medicini zbog biokompatibilnosti, dobrih mehaničkih svojstava i otpornosti na koroziju (1 – 3). Ida i suradnici prvi su ga počeli primjenjivati u protetici još sredinom osamdesetih godina prošlog stoljeća (4). I tada su, kao i danas, metal-keramički sustavi bili *zlatni standard* (5). Upotreba komercijalno čistog titanija za izradu osnovne konstrukcije metal-keramičkih radova izazov je zbog visoke temperature taljenja (od 1668 °C) i sklonosti stvaranja debeloga oksidnog sloja. Pri temperaturi >880 °C nastaje transformacija titanija iz α-faze u β-fazu i povećava mu se sklonost reagiranju s kisikom (6 – 8). Navedena svojstva titanija povećavaju cijenu takvih nadomjestaka i smanjuju vrijednosti vezne čvrstoće s keramikom u usporedbi s ostalim metalima za izradu osnovne konstrukcije, a to limitira njegovu širu primjenu (9 – 12).

Zato su se tražili novi proizvodni procesi kako bi taj biološki iznimno prihvatljiv materijal postao pristupačniji za ši-

Introduction

Commercially pure titanium (CP Ti) has been recognized in dentistry for its biocompatibility, good mechanical properties and corrosion resistance (1-3). Due to this, the application of CP Ti in prosthodontics dates back to the 1980s and the works of Ida et al (4). Metal ceramic restorations have been a golden standard both for long term aesthetic and functional restorations (5). Using CP titanium as a base material for metal ceramic systems has been a challenge due to its high melting temperature (1668 °C) and a tendency to form a thick oxide surface layer. This is due to the phase transformation of titanium form α to β structure at a temperature of 880 °C (6-8). These properties cause high manufacturing costs and lower bond strength values in comparison to other base metals used in metal-ceramic systems (9-12).

Powder metallurgy (P/M) is a series of manufacturing processes which involve mixing, pressing and heating of varroku upotrebu. Metalurgija praha (P/M) niz je proizvodnih procesa koji uključuju miješanje, tlačenje i zagrijavanje raznih praškastih metala i legura u željeni oblik. Na taj način snizila se cijena proizvoda od titanija i njegovih legura (13).

P/M Ti i njegove legure mogu se, prema pripremi praha, klasificirati u tri kategorije: već legirani, brzo skrućeni i miješani elementarni P/M Ti prah. Korištenje miješanoga elementnog titanija znatno je jeftinije zbog niske cijene i titanija i ostalih elementnih prašaka. Najčešće korišteni prašci su spužvasti titanij i hidrogenirani-dehidrogenirani (HDH) titanij (14). Najučinkovitije P/M tehnologije u izradi struktura od komercijalno čistoga titanija najčešće uključuju hladnu (CIP) i toplu (HIP) konsolidaciju praha te tople deformacijske procese. Pritom se istodobno povećavaju temperatura i tlak, što rezultira znatno gušćim komponentama u usporedbi sa sinteriranim materijalima ili čak s onima neporoznima (13, 14).

Erozimat sa žicom (WEDM) koristi se termoelektričnim procesom u kojemu se materijal uklanja s radnog tijela stroja primjenom toplinske energije – iskrenjem. Izboj električne energije ponavlja se između dviju elektroda (stroja i obrađivanog materijala) uronjenih u dielektrični fluid. Kako temperatura ispod elektrode raste zbog stvaranja iskre, materijal se počinje taliti i radna površina evaporira (15 – 17).

Konvencionalna strojna obrada komercijalno čistog titanija zahtjevna je zbog visokih temperatura i brzog trošenja alata za obradu (15). Zbog toga se takav titanij smatra materijalom *teškim za obradu,* pa se sve češće upotrebljavaju nekonvencionalni načini strojne obrade koji uključuju i rezanje žicom na erozimatu (16 – 18).

Čvrsta veza između osnovnog materijala i obložne keramike nužna je za funkcijsku trajnost metal-keramičkih nadomjestaka (19). Takva vezna čvrstoća između osnovne konstrukcije i obložne keramike može se ispitivati različitim testovima. Nekoliko autora predlaže smične testove (20 – 23). No kako Međunarodna organizacija za normiranje (ISO) predlaže trotočkasti savojni test, sva recentna istraživanja koriste se tom metodom za ispitivanje vezne čvrstoće (24 – 31). Trotočkasti savojni test prema Schwickerathu, koji je opisan u normi ISO 9693, propisuje veličinu uzoraka metala potrebnih za ispitivanje: (25 ± 1) mm × (3 ± 0,1) mm × (0,5 ± 0,05) mm (24). Uzorci se obično proizvode lijevanjem ili glodanjem komercijalno dostupnog titanija. Nakon proizvodnog procesa, u gotovo svim istraživanjima, uzorci su se dodatno brusili i polirali kako bi se postigle propisane dimenzije. Svrha ovog istraživanja bila je istražiti moguću primjenu WEDM-a u oblikovanju uzoraka za ispitivanje vezne čvrstoće prema normi ISO 9693 i utjecaj te tehnologije na mikrostrukturu i sastav rezne površine uzoraka P/M CP Ti.

Materijali i metode

Materijal CP Ti proizveden je korištenjem Ti 99,4 % HDH praha veličine čestice <150 µm. U prvoj fazi korišten je CIP i tlak od 200 MP-a na sobnoj temperaturi. Nakon toga materijal je podvrgnut toplom prešanju u vakuumu (HVP) na temperaturi od 420 °C i tlaku od 330 MP-a.

ious powdered metals and alloys into a desired shape. Powder metallurgy has been taken into account for lowering the cost of titanium parts (13). P/M titanium and its alloys can be classified, according to the adoption of raw powder, into three categories: pre-alloyed P/M Ti alloys, rapid solidified P/M Ti alloys, and blended elemental P/M Ti alloys. Use of blended elemental powder is much more cost-effective, due to cheap Ti and other elemental powders. Commonly used titanium powder includes sponge Ti fines and the hydrogenation and dehydrogenation (HDH) Ti powder (14). Among the powder metallurgy techniques, cold (CIP) and hot (HIP) isostatic pressure and hot deformation processes of metallic powder, which involve the simultaneous application of pressure and temperature, result in engineering components with higher relative density compared to the conventional sintering, or even fully dense materials (13,14).

Electric discharge machining EDM utilizes a thermo electrical process in which material is removed from work piece by applying the heat energy of sparks. Electrical discharge is repeated between two electrodes (tool and work piece) in the presence of a dielectric fluid. The temperature of the area under spark increases. As a result, the materials melt and vaporize from localized area by using spark energy (15-17).

Machining CP Ti by conventional machining methods has some disadvantages such as high cutting temperature and high tool wear ratio (15). Thus, CP Ti is classified as "difficult-to-machine" material. Therefore, unconventional machining processes, including wire EDM (WEDM) are introduced for machining CP Ti (16-18).

Strong bonding to porcelain is essential for long term success of metal ceramic restorations (19). Bond strength between substructure metals and veneering ceramics can be tested using various methods. Several authors have proposed the use of the shear bond test for strength evaluation (20-23). However, since the International Organization for Standardization (ISO) standard for metal ceramics advocates a three-point bending test, almost all recent studies have used this method to evaluate bond strength (24-31). The three point bending test according to Schwickerath, described in the ISO 9693, requires metal samples with dimensions (25 ± 1) mm × (3 ± 0.1) mm × (0,5 ± 0.05) mm (24). So far, casting and milling of commercially available CP Ti has been used for manufacturing of such samples. Also, almost all of the studies found used grinding and polishing of the samples after manufacturing to obtain the required dimensions. The aim of the present study was to evaluate the effects of WEDM manufacturing process on the surface quality of the P/M CP Ti samples manufactured for ISO 9693 bond strength testing.

Materials and methods

CP Ti material was prepared using Ti 99.4% HDH powder with particle size <150 µm. In first step CIP was used at 200 MPa at room temperature, later hot vacuum pressed at 420 °C and 330 MPa and finally consolidated by direct extrusion at 500 °C to 13 x 13 mm bars using reduction area ratio

Završna konsolidacija obavljena je izravnom ekstruzijom na temperaturi od 500 °C, koristeći se omjerom redukcije površine od 1 : 4,2. Proizvedene su tri šipke dimenzija 150 x 13 x 13 milimetara. Uzorci za ISO 9693 proizvedeni su od tih šipki tehnologijom WEDM.

Za istraživanje je bilo pripremljeno ukupno osam uzoraka i podijeljeni su u dvije grupe. Prvoj nakon WEDEM-a površina nije bila obrađena, a druga je bila metalurški pripremljena silicij-karbidnim brusnim papirima gradacije P 320 – P 4000 (Struers Inc., West Lake, SAD). Površine obiju grupa analizirane su metodama SEM, EDS i XDR.

SEM i EDS

Nakon izrezivanja uzoraka P/M CP Ti s pomoću WEDM-a, rezna površina analizirana je metodama SEM i EDS u poprečnom presjeku. Uzorci su bili istovjetno pripremljeni – izrezani su, pohranjeni u polimerni materijal i brušeni. SEM analiza obavljena je mikroskopom VEGA TESCAN TS5136LS u visokom vakuumu s naponom od 20 keV. Mikroanaliza kemijskog sastava obavljena je detektorom Oxford EDS, a područje analize bilo je 20 keV. Površina uzorka analizirana je prije i poslije brušenja.

Rendgenska difrakcijska analiza (XDR)

Uzorci su ispitivani na sobnoj temperaturi metodom rendgenske difrakcije u polikristalu koristeći se Philipsovim difraktometrom PW 1830 sa zračenjem CuKα. Podtaci su se prikupljali u području 2θ kuta između 10 i 100°, u koraku 0,02° te s vremenom prikupljanja od jedne sekunde po koraku. Kvalitativna i kvantitativna fazna analiza provedene su s pomoću Rietveldova utočnjavanja koristeći se programom HighScoreXPert Plus. Za opisivanje pozadinskog šuma primijenjen je polinomialni model, a za opisivanje difrakcijskih profila pseudo-Voightova funkcija. Pomak ljestvice, čimbenik skaliranja, parametri poluširine (U, V, W) i asimetrije te parametri profilnoga oblika istodobno su utočnjavani. Također su utočnjavani i strukturalni parametri, parametri jedinične ćelije te atomski temperaturni čimbenici. S pozicijama atoma to se nije činilo jer se svi nalaze na kristalografski specijalnim položajima.

Rezultati

Rendgenska difrakcijska strukturna analiza u polikristalu (XRPD)

Dvije skupine po četiri uzorka, neobrađeni uzorci (označeni kao S 1) i brušeni uzorci (označeni kao S 2) analizirani su rendgenskom difrakcijom u polikristalnom materijalu (XRPD). Grafički prikaz Rietveldova utočnjavanja za obje skupine uzoraka nalazi se na slici 1.

Rietveldovo utočnjavanje provedeno je na temelju početnih strukturnih modela iz ICSD-baze podataka: 28955

1:4.2. A total of 3 bars dimensions 150 x 13 x 13 mm were manufactured. From these bars samples were manufactured in the dimensions proposed by ISO 9693 using wire EDM. The basic parts of the WEDM machine consist of a wire electrode, a work table, and a servo control system, a power supply and dielectric supply system. In this study, copper wire of 0.25 mm diameter was used as a tool and distilled water was used as a dielectric fluid. The other input parameters important for surface quality after cutting were gap voltage (40 V), wire feed (5m/min), pulse on time (5s), and pulse off time (24s). These parameters remained constant for all samples during WEDM.

Eight samples were made and divided into 2 groups. The first group was untreated after WEDM, and the second was ground by use of silicon carbide papers P320-P4000 (Struers Inc., West Lake, US). Both groups were analysed using SEM, EDS and XDR.

SEM and EDS analyses

After WEDM, cutting surfaces were analysed in cross sections by use of SEM and EDS equipment. For these analyses all samples were prepared under the same parameters and conditions: cut, mounted, ground and polished. The analyses were made by use of scanning electron microscope VEGA TESCAN TS5136LS in high vacuum with voltage of 20kV. Microanalysis of chemical composition was made with Oxford EDS detector, while the area of analysis of detector was 20keV. The sample surface was analysed before and after grinding.

X-ray structural analysis

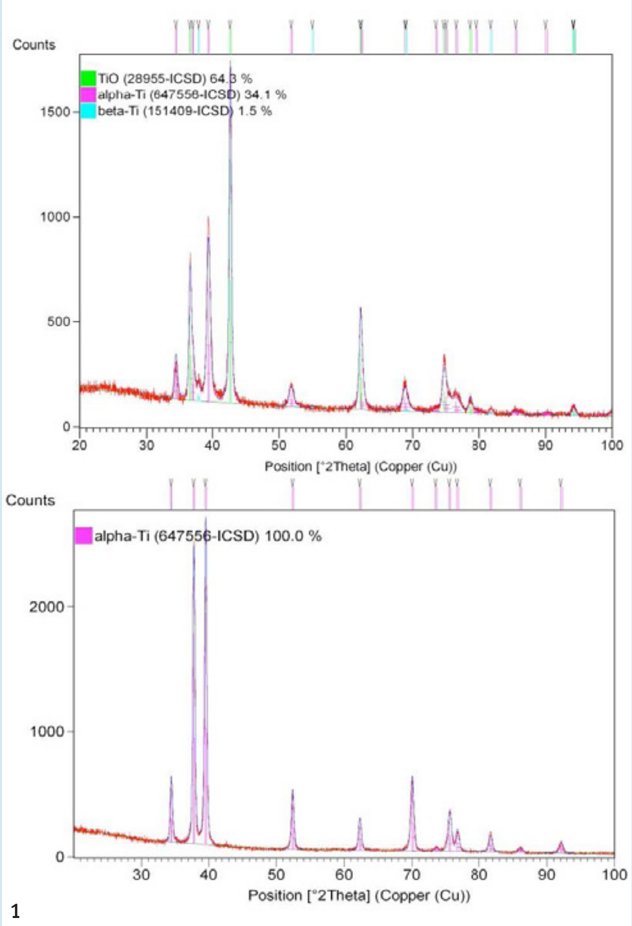
The samples were studied at room temperature by X-ray diffraction (XRD) analysis using a Philips PW 1830 diffractometer with CuKα radiation. Data were collected over a 2θ range between 10 and 100° 2θ in a step scan mode with steps of 0.02° and counting time of 1 s per step. Qualitative and quantitative phase analysis was performed by means of Rietveld refinement for HighScore XPert Plus program. Polynomial model was used to describe the background. Pseudo-Voigt function was used to describe diffraction profiles. During the refinement, a zero shift, scale factor, half-width parameters (U, V, W), asymmetry parameters and peak shape parameters were simultaneously refined. Also, the structural parameters, unit-cell constants and temperature factors were refined. Atomic position was not refined since all atoms are located in special positions.

Results

X-ray structural analysis

Two samples, a prepared sample (denoted S1) and a ground one, denoted S2, were analysed by X-ray powder diffraction (XRPD). Graphical result of Rietveld refinement for both samples is shown in Figure 1.

Rietveld refinements were carried out by using starting structural models from ICSD data base: 28955, 647556 and 151409 for TiO, α-Ti and β-Ti, respectively. Quantitative and qualitative analysis of sample S1 revealed that sam-



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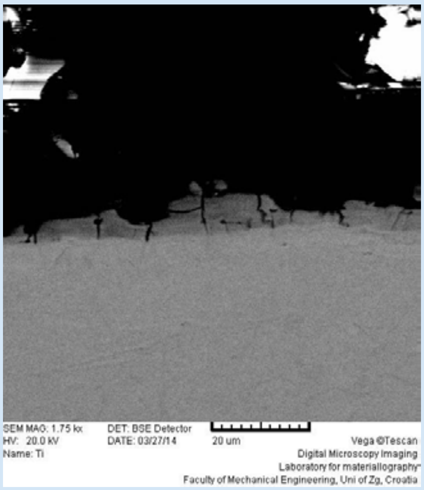

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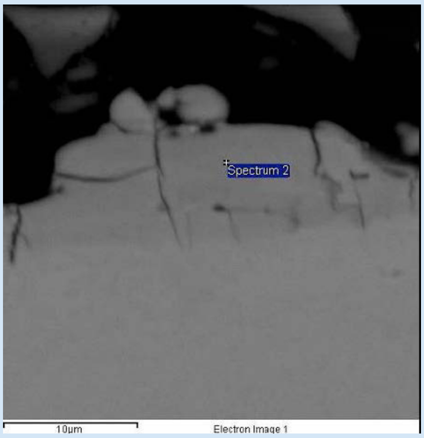

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Slika 1. Grafički prikaz rezultata završnoga Rietveldova utočnjavanja podataka na sobnoj temperaturi za uzorke S 1 (gore) i S 2 (dolje); eksperimentalni podatci prikazani su crvenom crtom, izračunata difrakcijska slika plavom, a zelene, magenta i tirkizne okomite oznake difrakcijski su položaji za kristalne faze TiO, α-faze i β-faze titanija

Figure 1 Graphical presentation of the final Rietveld refinement of room temperature data for samples S1 (up) and sample S2 (down). Experimental data are shown in red, the calculated pattern is blue while vertical marks, green, magenta and turquoise, represent the diffraction reflections for crystal phases TiO, α-Ti and β-Ti, respectively.

Slika 2. Presjek površine P/M CP Ti analiziran SEM-om

Figure 2 SEM cross section surface of titanium samples machined by WEDM

Slika 3. Detalj osnovnoga materijala s površinskim slojem i pozicija EDS analize

Figure 3 SEM cross section and location of EDS analysis on surface layer

(TiO), 647556 (α-fazu Ti) i 151409 (β-fazu Ti). Kvantitativna i kvalitativna analiza uzorka S 1 pokazala je da sadržava TiO kao dominantu fazu u težinskom udjelu na površini uzorka (64,3 %), α-Ti faza prisutna je u manjem udjelu (34,1 tež. %), a β-Ti faza nalazi se samo u tragovima (1,5 tež. %). Difrakcijska slika uzoraka S 2 pokazuje drastičnu razliku u faznom sastavu, što je posljedica mehaničke obrade površine. Uzorak S 2 potpuno je jednofazan i sadržava samo titanijevu α-fazu (100 tež. %) Faza α-Ti kristalizira se u heksagonskoj kristalnoj rešetki u prostornoj grupi p 63/mmc s utočnjenim parametrima jedinične ćelije *a* = 2,9555(2) i *c* = 4,6893(3).

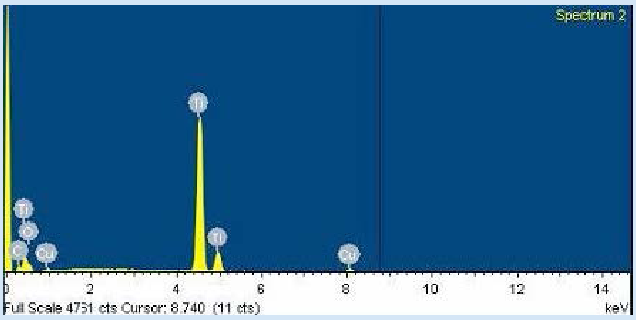
SEM i EDS

Na slici 2. poprečni je presjek uzorka P/M CP Ti nakon rezanja WEDM-om. Vidi se osnovni materijal prekriven slojem različitog sastava koji se prostire po cijeloj reznoj površini. U tom sloju nalaze se mnogobrojne frakture usmjerene s površine prema unutrašnjosti materijala (slika 3.). Rezultati EDS analize tankog sloja na reznoj površini uzoraka P/M CP

ple contained TiO as dominant phase (64.3 wt%), α-Ti was present in smaller amount (34.1 wt%) while β-Ti can was present only in traces (1.5 wt%). XRD pattern of sample S2 (obtained by mechanical treatment of S1) showed that its composition changed drastically upon mechanical treatment. Sample S2 was a completely single-phased sample containing only α-Ti (100 wt%) and no additional phases present. α-Ti crystallized in hexagonal crystal system, in space group P 63/mmc with unit-call parameters a= 2.9555(2) and c=4.6893(3).

SEM and EDS analyses

Typical surface of P/M CP Ti samples machined by WEDM in cross section is shown in Figure 2. The base material of sample is covered by thin layer of material with different composition, arrow in Figure 2. In this layer a lot of fractures are visible, directed from the surface of material to the base (Figure 3). The results of EDS analysis of thin lay-



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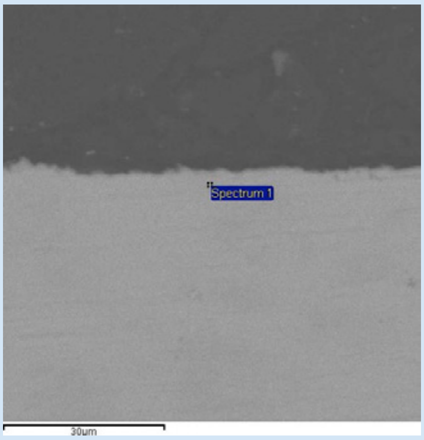

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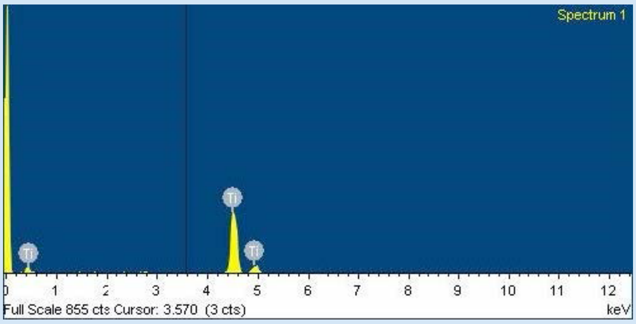

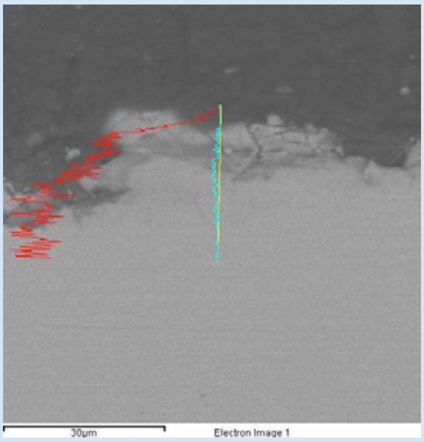

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Slika 4. Rezultati EDS mikroanalize kemijskog sastava površinskog sloja prikazanog u spektru
Figure 4 Results of EDS microanalyses of the layer's chemical composition are presented in spectrum
Slika 5. Presjek površine P/M CP Ti analiziran SEM-om i pozicija EDS analize
Figure 5 SEM cross section and location of EDS analysis after grinding
Slika 6. Rezultati EDS mikroanalize kemijskog sastava površinskog sloja prikazanog u spektru
Figure 6 Results of EDS microanalysis of surface layer's chemical composition after grinding are presented in spectrum
Slika 7. Linijska EDS analiza presjeka nebrušenog uzorka P/M CP Ti
Figure 7 Line analysis of surface layer of untreated samples with concentrations of Ti and Cu

Ti prikazani su spektrom na slici 4. i pokazuju da se sloj sastoji od Ti, Cu, C i O.

Površina uzorka nakon brušenja i uklanjanja 20 µm materijala prikazana je na slici 5. U presjeku se vidi homogeni materijal bez pukotina. Rezultati EDS analize pokazuju da se materijal sastoji od titanija bez oksida (slika 6.). Zbog boljeg prikaza obavljena je linijska EDS analiza koncentracije titanija i bakra u presjeku nebrušenog uzorka (slika 7.).

Rasprava

Kvalitetna veza komercijalno čistog titanija s dentalnom keramikom težak je i osjetljiv postupak. Kako bi se postigla, vrlo su važni sastav i struktura površine titanija. Dobri rezultati vezne čvrstoće postižu se samo s α-fazom titanija i što tanjim oksidnim slojem (11). U prvom dijelu ovoga istraživanja uzorci P/M CP Ti proizvedeni tehnologijom WEDM imaju neodgovarajuću površinu. Mnoštvo pukotina te drugi elementi i spojevi poput bakra i oksida, u površinskom sloju mogu kompromitirati vezu između titanija i dentalne ke-

er on cutting surface of Ti samples are presented in Figure 4 and show that the layer consists of Ti, Cu, C and O probably titanium and copper oxides and some carbide. The surface area of samples after grinding and removal of 20µm of surface material is shown in Figure 5. The Figure shows uniform composition of material and without visible fractures. The results of EDS analysis show a high level of pure Ti with no oxides (Figure 6). For better illustration purposes, the line of EDS analysis of Ti and Cu concentrations in cross section of untreated samples is shown in Figure 7.

Discussion

Bonding of CP Ti to dental ceramics is a difficult and sensitive process. Titanium surface structure and composition are crucial to establishing a good bond. Only a pure α-phase structure with a thin oxide layer is acceptable to obtain good bonding strength results (11). In the first part of this study, the WEDM manufactured samples of P/M CP Ti showed an inadequate surface layer. The layer had a large number of fractures and the composition showed traces of other elements (Cu) and oxides which could compromise ramike. Frakture su vjerojatno nastale zbog unutarnjeg naprezanja uzrokovanog velikim temperaturnim gradijentom na površini materijala koji nastaje tijekom proizvodnje. Te su pukotine opasne tijekom korištenja materijala jer pri dinamičkom opterećenju svako oštećenje potiče propagaciju, širenje pukotine. Pronalazak bakra u tankom površinskom sloju može se također objasniti visokom temperaturom tijekom proizvodnje i reakcijom između bakrene žice uređaja WEDM-a i površine uzorka. Zbog visoke reaktivnosti s kisikom titanijeva β-faza brzo na površini stvara oksidni sloj. Velika količina oksida pronađena na površini može se pripisati temperaturi (>880 °C) koja je viša od transformacije titanija iz α-faze u β-fazu. U dosadašnjim istraživanjima ističe se da zbog nastanka debeloga oksidnog sloja na površini titanija nastaju adhezivni i kohezivni lomovi između titanija i dentalne keramike (7, 8, 11, 25 – 31).

Nakon brušenja se znatno poboljšala kvaliteta površine za ostvarivanje veze s dentalnom keramikom uzoraka. Homogena površinska struktura bez oksida i drugih nečistoća prikladna je za kvalitetnu vezu s keramikom. Utjecaj visoke temperature ograničen je na tanki površinski sloj (10 µm). Poboljšanje kvalitete površine nakon uklanjanja površinskog sloja od 20 µm može se objasniti odgovarajućim hlađenjem tijekom WEDM-procesa i niske toplinske vodljivosti titanija (10). Također je jasno da je WEDM izvrstan za proizvodnju uzoraka titanija za ispitivanje vezne čvrstoće prema normi ISO 9693, uz naknadno brušenje i uklanjanje tankoga površinskog sloja.

Zaključak

Unutar ograničenja ovog istraživanja može se zaključiti:

1. XDR analiza WEDM-om proizvedenih i neobrađenih uzoraka pokazuje značajni udjel oksida, titanijeve β-faze i drugih nečistoća na površini. Nakon što je s uzorka brušenjem uklonjen tanki površinski sloj, pronađen je titanij u α-fazi, što je nužno za kvalitetnu vezu s dentalnom keramikom.
2. SEM i EDS analize WEDM-om proizvedenih nebrušenih uzoraka pokazuju da u tankom površinskom sloju ima bakra i oksida. Tijekom proizvodnje, zbog visoke temperature unutar površinskog sloja, nastaju pukotine s površine prema unutrašnjosti uzoraka. Te pukotine opasne su tijekom korištenja materijala jer pri dinamičkom opterećenju svako oštećenje može potaknuti njihovu propagaciju. Nakon brušenja uzoraka i uklanjanja tankoga površinskog sloja, vidi se homogena struktura čistog titanija bez pukotina.
3. Proizvodni proces WEDM može biti korišten u proizvodnji uzoraka komercijalno čistoga titanija za ispitivanje vezne čvrstoće prema normi ISO 9693, ali samo uz naknadno brušenje površine.

Zahvale

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the bond between titanium and dental ceramics. The cause of these fractures was most probably internal stress caused by very high temperature gradient in material during the WEDM. Also, the findings of Cu in the thin surface layer can be explained with the high temperature and the reaction between the cooper wire of the WEDM machine and the sample surface. High levels of oxides are also attributed to the high temperature which is higher (>880 °C) than the titanium phase transformation from α to β. The finding of C is attributed to sample preparation. XDR analysis of the untreated samples showed a high level of titanium oxides on the surface and a smaller amount of α and trace amounts of β-phase titanium. Because of its high reactivity with oxygen β-phase, titanium quickly forms a thick oxide layer on the surface. It can be assumed that all of the titanium oxide that was found on the surface of the samples came from the large amount of β-phase titanium. Several studies emphasised that β-phase and its reaction to form a thick oxide layer compromised the bond and caused adhesive and cohesive fractures between titanium and dental ceramics (7, 8,11,25-31).

After grinding the surface, quality significantly improved. Analysis showed no oxides or other impurities which presented a suitable surface for ceramic bonding. The effects of high temperature are limited to a thin layer on the surface (10 µm) (Figure 3). Such an improvement after removal of a 20 µm layer thickness of material (Figure 5) can be explained by the adequate cooling during the WEDM process and low heat conductivity of titanium (10). It also shows that WEDM is an excellent method of machining samples for ISO 9693 if accompanied by grinding and removing a thin surface layer.

Conclusion

Within the limitations of this study it can be concluded:

1. XDR analysis of WEDM manufactured untreated samples showed high levels of oxide formationβ-phase titanium and impurities on the surface. After grinding and removal of the thin surface layer, pure α-phase titanium was found which is essential for adequate bond strength to dental ceramics.
2. SEM and EDS analyses of WEDM manufactured untreated samples showed traces of impurities (Cu) and oxides in the thin surface layer. Fractures found within the surface layer of the samples and directed from the surface to the base of material developed due to the high temperature gradient during manufacturing. After grinding and removal of the surface layer, a homogenous structure of pure titanium with no fractures was visible.
3. WEDM manufacturing can be used for producing titanium samples for ISO 9693 but only if accompanied with surface grinding.

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Izjava

Nema sukoba interesa.

Conflict of interest

None to declare

Abstract

Purpose: Commercially pure titanium (CP Ti) has been recognized in dentistry for its biocompatibility, good mechanical properties and corrosion resistance. Conventional manufacturing processes can affect surface quality and result in poor bonding of dental ceramics to CP Ti. This is why powder metallurgy (P/M) and wire electro-discharge machining (WEDM) are being introduced in the manufacturing process. The aim of this study was to evaluate the effect of WEDM on the surface composition and microstructure of P/M CP Ti samples produced for bond strength testing according to ISO 9693. **Materials and methods**: Eight samples of P/M CP Ti, dimensions according to ISO 9693, were made using WEDM and divided in two groups (untreated and grinded). Microanalyses of chemical composition and microstructure of both groups were made using SEM, EDS and XDR. **Results:** SEM and EDS analysis of untreated samples showed a thin layer on surfaces with fractures in it. Grinded samples showed homogenous structure with no layer and no fractures. XDR analysis showed high level of oxides on the surface of untreated samples, while after grinding only pure α-phase was found. **Conclusion**: WEDM is a suitable method of sample production for ISO 9693 if accompanied by grinding with silicon carbide papers P320-P4000.

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