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Ispitivanje povezanosti hrapavosti površine cirkonij-oksidne keramike i vezne čvrstoće s obložnom keramikom: prethodno priopćenje

Correlation between Surface Roughness and Shear Bond Strength in Zirconia Veneering Ceramics: a Preliminary Report

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Sažetak
Delaminacija i *chipping* obložne keramike opisani su kao najčešći uzroci za neuspjeh terapije cirkonij-oksidnim nadomjescima. **Svrha:** Željela se ustanoviti povezanost između hrapavosti površine jezgre od cirkonijeva oksida i vezne čvrstoće obložne keramike. **Materijali i postupci:** Sinterirano je ukupno 10 uzoraka koji su bili podijeljeni u dvije skupine. Uzorci prve skupine polirani su, a oni iz druge polirani i pjeskareni. Nakon obrade površine cirkonij-oksidne jezgre napečena je obložna keramika debljine tri milimetra. Tako pripremljeni uzorci uloženi su u PTFE-kalupe s pomoću smole PMMA i izloženi smičnoj sili do pucanja (univerzalna kidalica). Hrapavost površine izmjerena je prije napečenja obložne keramike instrumentom s ticalom Perthometer S8P, te su zabilježene vrijednosti Ra i Z. **Rezultati:** Polirani i pjeskareni uzorci imali su znatno hrapaviju površinu ($p<0,01$) te su pokazali mnogo više vrijednosti vezne čvrstoće ($p<0,01$) od samo poliranih uzoraka. **Zaključak:** Utvrđena je značajna povezanost između vrijednosti hrapavosti površine cirkonij-oksidne jezgre i vezne čvrstoće ($r=xy$, $p<0,01$) s obložnom keramikom.

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Ključne riječi
stomatološke ljuske; cirkonij-oksid; keramika; svojstva površina; smična sila; dentalni cementi;

Uvod

Krunice i mostovi višeslojni su objekti složene geometrije (1 – 3). Tradicionalno nastaju slojevanjem obložne keramike sloj po sloj na čvrsti jezgreni materijal (4,5). Strukturni integritet spoja između obložne keramike i jezgrenog materijala kritični je čimbenik koji utječe na trajnost potpuno keramičkog nadomjeska. Zato su delaminacija i *chipping* opisani kao najčešći uzroci neuspjeha u terapiji tim nadomjescima (6). Učestalost loma obložne keramike znatno je veća kod cirkonij-oksidne keramike u usporedbi s metal-keramičkim sustavima (7). Kvaliteta spoja između cirkonijeva oksida i obložne keramike do danas je nerazjašnjena. Veznu čvrstoću određuju mnogobrojni čimbenici – obrada površine jezgrenog materijala, snaga kemijskih veza, mehanička povezanost, vrsta i koncentracija defekata na međuspoju (8) te svojstvo vlaženja površine i stupanj naprezanja u sloju obložne keramike zbog razlike u koeficijentu termičke istezljivosti (KTI) jezgre i obložne keramike (9). U stomatologiji se najčešće rabi itrijem stabilizirani cirkonijev oksid (Y-TZP) visoke čvrstoće s koncentracijom kristalne faze većom od 90 posto (10).

Introduction

Dental crowns and bridges are generally laminar structures, with complex geometries (1-3). Traditionally, esthetic porcelain is veneered onto a strong core material layer by layer. In the case of all-ceramic restorations, the core material is usually zirconium (4, 5). The structural integrity of the interface between porcelain veneer and zirconia core is a critical factor in the longevity of all-ceramic dental crowns and fixed dental prostheses. That is why delamination and chipping of veneering porcelain were described as the most frequent reasons for the failure (6). The incidence of veneer fractures in zirconia FPDs was significantly higher compared to the metal-ceramic FPDs (7).

There is not much reliable information available about the bond quality between veneering ceramics and zirconia core. Bond strength is determined by many factors: surface finish of the core, strength of chemical bonds, mechanical interlocking, type and concentration of defects at the interface (8), wetting properties and the degree of compressive stress in the veneering layer due to difference in the coefficients of

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Zato Y-TZP nije prikladan za jetkanje (11), pa se za obradu njegove površine primjenjuju druge metode kao, primjerice, pjeskarenje česticama aluminijeva oksida (12 – 15). Neka istraživanja pokazala su znatan utjecaj pjeskarenja i korištenja veznog sredstva na veznu čvrstoću (16 – 18). Tim načinima obrade smanjila se hrapavost površine i nastala su potkopana područja (19 – 22). No, pjeskarenje također inicira transformaciju zrna na površini te time utječe i na kvalitetu veze s obložnom keramikom (23,24). KTI monoklinskih zrna cirkonijeva oksida iznosi 7,5×10–6/°C, a KTI tetragonskih zrna je 10,8×10–6/°C. U skladu s tim povećanje razlike u KTI-u smanjuje veznu čvrstoću cirkonijeva oksida s obložnom keramikom (25). Zato je utjecaj pjeskarenja na mehanička svojstva Y-TZP-a, te na veznu čvrstoću s obložnom keramikom, još uvijek predmet mnogih istraživanja (23,26,27). Smični test uobičajeni je način mjerenja vezne čvrstoće među slojevima kod potpuno keramičkih sustava (10,28 – 33).

Svrha istraživanja bila je ustanoviti povezanost između hrapavosti površine cirkonijeva oksida i vezne čvrstoće s obložnom keramikom.

Materijali i postupci

U istraživanju su korišteni blokovi presinteriranog cirkonijeva oksida IPS e.max ZirCAD (Ivoclar Vivadent AG, Schaan, Liechtenstein) podijeljeni u dvije skupine po pet uzoraka. Sinteriranjem u peći Programat S1 (Ivoclar Vivadent AG, Schaan, Liechtenstein) šest sati na temperaturi od 1500° C blokovi su dostigli dimenzije 10 x 10 x 10 milimetara potrebne za istraživanje, pa glodanje nije bilo potrebno. Slijedilo je poliranje svih uzoraka dijamantnim svrdlima finoće 90 μm (Komet, Salzburg, Austrija) pod vodenim mlazom s maksimalnim brojem okretaja (200 000 rpm) i minimalnim pritiskom kako bi se osiguralo jednolično poliranje površine. Uzorci su očišćeni 70-postotnim etanolom, zatim deset sekundi vodenom parom te osušeni zrakom. Pet uzoraka podvrgnuto je mjerenju hrapavosti površine i na njih je napečena obložna keramika, a ostalih pet je naknadno pjeskareno (Renfert duo pro, GmbH, Hilzingen, Njemačka) česticama aluminijeva oksida (110 μm) (Cobra; Renfert, GmbH, Hilzingen, Njemačka) pod tlakom od 2,5 bara u trajanju od pet sekundi, očišćeno je parom, osušeno zrakom te ostavljeno za analizu površine i napečenje keramike.

Hrapavost površine izmjerena je instrumentom s ticalom Perthometer S8P (Perthen, Mahr, Göttingen, Njemačka). Pri mjerenju ticalo se kretalo po površini konstantnom brzinom i silom mjerenja od 1,3 mN. Mjerenja su obavljena Gaussovim filtrom s *cut-off* vrijednošću od λc = 0,8 milimetara i duljinom mjerenja od ln=5,6 milimetara. Srednja hrapavost (Ra) i Z- vrijednosti (udaljenost između najviše i najniže toč-

thermal expansion between zirconia and veneering ceramics (9). In contrast to conventional dental ceramics, the zirconia systems currently available for use in dentistry are high strength Y-TZP with a zirconium dioxide crystalline content higher than 90% (10). For this reason, Y-TZP ceramics are not suitable for acid etching (11), therefore, other surface treatment methods such as airborne particle abrasion with aluminum oxide particles (Al_2O_3) are used (11-15).

Some studies showed that the bonding strength and the mode of failure were significantly affected by surface treatments such as use of liners and sandblasting which increased the surface roughness and provided undercuts (16-22). However, sandblasting also initiates phase transition, thus affecting mechanical strength and most likely, the bonding capacity of the veneering material (23, 24). The coefficient of thermal expansion of monoclinic zirconia is 7.5×10–6/°C, and that of tetragonal zirconia is 10.8×10–6/°C. Accordingly, an increase in the difference in the coefficient of thermal expansion between the TZP framework and the veneering ceramic leads to a decrease in bond strength (25). Therefore, the effect of sandblasting on the mechanical strength of Y-TZP and the bond quality to veneering ceramics is an intensely discussed subject (23, 26, 27). To measure the bond strength of all-ceramic systems, shear tests are generally used to evaluate the influence of the core surface on bond quality (10, 28-33).

The aim of the study was to investigate the correlation between zirconia surface roughness and shear bond strength of veneering ceramics.

Materials and methods

Two groups of pre-sintered Y-TZP IPS e.max ZirCAD blocks (Ivoclar Vivadent AG, Schaan, Liechtenstein) were used in the study, each group consisting of 5 specimens. By sintering to full density in the Programat S1 furnace (Ivoclar Vivadent AG, Schaan, Liechtenstein) during 6 hours at the temperature of 1500°C, the blocks gained dimensions of 10x10x10 mm, so CAD/CAM grinding was not necessary. All samples were afterwards ground under water spray jet incorporated in the hand-piece with a 90μm grit diamond bur (Komet, Salzburg, Austria) at the maximum revolutions (200,000 rpm) and with minimal pressure to ensure a consistent grinding speed. The cubes were cleaned with 70% ethanol, and then steam cleaned for 10 seconds and air dried. Five samples were left for surface roughness analysis and veneering, and other five samples were additionally sandblasted (Renfert duo pro, GmbH, Hilzingen, Germany) with 110 μm Al_2O_3 particles (Cobra; Renfert, GmbH, Hilzingen, Germany) at 2.5 bar pressure for a period of 5 seconds, steam-cleaned, air-dried and also left for surface roughness analysis and veneering.

The measurement of surface roughness was performed using the Stylus instrument Perthometer S8P (Perthen, Mahr, Göttingen, Germany). During the measurement the stylus moved at a constant speed across the samples with a measuring force of 1.3 mN. Measurements were performed by using Gauss filter with cut-off value of λc = 0.8 mm and the evaluation length ln = 5.6 mm. Mean roughnes (Ra) and Z values (the distance from the highest to the lowest point of ke na mjerenoj površini) zabilježene su za svaki uzorak te su statistički obrađene.

Na uzorke je tada tehnikom slojevanja napečena obložna keramika IPS e.max Ceram (Ivoclar Vivadent AG, Schaan, Liechtenstein). Kako bi se postigle odgovarajuće dimenzije obložne keramike (10 x 5 x 3 mm), bila su potrebna dva pečenja u peći Programat P700 (Ivoclar Vivadent AG, Schaan, Liechtenstein).

Test vezne čvrstoće proveden je univerzalnom kidalicom (model LRX, Lloyd Instruments, Fareham, Velika Britanija). Svaki je uzorak uložen u PTFE-kalupe s pomoću smole PMMA (Struers Co, Ballerup, Danska). Uzorci su postavljeni u kidalicu tako da je spoj dvaju materijala bio u razini njezina klina. Smična sila bila je usmjerena na spojnu površinu brzinom od jednog milimetra u minuti do loma koji se automatski bilježio računalom spojenim na kidalicu (Nexygen, Lloyd Instruments, Fareham, Velika Britanija).

Dobiveni podaci obrađeni su statističkim paketom SPSS (verzija 17,0, SPSS Inc., Chicago, IL, SAD). Nezavisnim t-testom ispitane su razlike među uzorcima. Piersonov koeficijent korelacije izračunat je između vrijednosti hrapavosti površine (Ra i Z) i vrijednosti vezne čvrstoće. P-vrijednosti manje od 0,05 smatraju se statistički značajnima.

measurement along the observed line) were recorded for each sample and were statistically analysed.

Samples were then veneered using a layering technique. The used veneering ceramic was IPS e.max Ceram (Ivoclar Vivadent AG, Schaan, Liechtenstein). Two separate firings in a firing furnace Programat P700 under the same conditions were required to establish the correct dimensions of the veneering ceramics (10x5x3mm).

Each sample was then embedded in the customized polytetrafluoroethylene (PTFE) moulds using the PMMA resin AcryFix (Struers Co, Ballerup, Denmark) to allow shear bond testing in the universal testing machine (model LRX, Lloyd Instruments, Fareham, Great Britain), with the core-veneer interface positioned at the level of the jig. Shear force was applied as close as possible to the veneer-core interface at a crosshead speed of 1 mm/min until fracture occurred, which was automatically registered and displayed by the computer software of the testing machine (Nexygen, Lloyd Instruments, Fareham, Great Britain).

Statistical analysis was performed using SPSS statistical package (version 17.0, SPSS Inc., Chicago, IL, USA). Independent samples *t*-test was used to test the difference between samples. Pierson's coefficient of correlation was calculated between surface roughness values (Z and Ra value) and the shear bond strength values. P value of less than 0.05 was considered statistically significant.

Rezultati

Rezultati (tablice 1, 2 i 3) su pokazali značajan utjecaj hrapavosti površine na veznu čvrstoću s obložnom keramikom. Nakon smičnog testa analiza loma pokazala je da je obložna keramika napečena na drugu skupinu uzoraka (polirani i pjeskareni) ostala vezana za površinu jezgrenog materijala pod znatno većim vrijednostima smične sile.

Results

According to the results (Tables 1, 2 i 3), surface roughness showed significant influence on the bond strength of the veneering ceramics. After the shear bond strength test, the results of the fracture analysis showed that in the second group (ground and sandblasted samples) the veneering ceramic remained on the zirconia surface under significantly higher shear forces.

Tablica 1. Deskriptivna statistika i značajnost razlike vrijednosti vezne čvrstoće (N) između poliranih te poliranih i pjeskarenih uzoraka
Table 1 Descriptive statistics and a significance of the difference for shear bond strength values (N) between ground, and ground and sandblasted samples

Obrada • ZrO_2 core treatment		N	X	SD	t	df	P
Vezna čvrstoća (N) • Shear bond strength (N)	Brušeni • Ground	5	264	55.5	- 5.89	8	<0.01
	Brušeni i pjeskareni • Ground and sandblasted	5	450	44.1			

N = broj uzoraka • number of samples; x = srednje vrijednosti • mean values; SD = standardna devijacija • standard deviation; t = t vrijednost • t value; df = stupanj slobode • degree of freedom; p = p vrijednost • p value

Tablica 2. Deskriptivna statistika i značajnost razlike Z- i Ra-vrijednosti (μm) hrapavosti površine između samo poliranih te poliranih i pjeskarenih uzoraka
Table 2 Descriptive statistics and a significance of the difference for surface roughness values (Z values) (μm) and Ra values (mean roughness) (μm) between ground and ground and sandblasted samples

Obrada • ZrO_2 core treatment		N	X	SD	t	df	P
Z vrijednost (μm) • Z value (μm)	Brušeni • Ground	5	4.03	0.16	- 8.84	8	<0.01
	Brušeni i pjeskareni • Ground and sandblasted	5	5.48	0.33			
Srednja hrapavost (Ra) (μm) • Mean roughness (Ra) (μm)	Brušeni • Ground	5	0.77	0.04	- 4.69	8	<0.01
	Brušeni i pjeskareni • Ground and sandblasted	5	0.95	0.07			

N = broj uzoraka • number of samples; x = srednje vrijednosti • mean values; SD = standardna devijacija • standard deviation; t = t vrijednost • t value; df = stupanj slobode • degree of freedom; p = p vrijednost • p value

Tablica 3. Korelacija između vezne čvrstoće (N) i vrijednosti hrapavosti površine (µm)
Table 3 Correlation between shear bond strength (N) and surface roughness values (µm)

		Z vrijednost • Z value (µm)	**Srednja hrapavost • Mean roughness (Ra) (µm)**
Vezna čvrstoća (N) • Shear bond strength (N)	Pearsonova korelacija • Pearson Correlation	0.88^{**}	0.88^{**}
	P (2-tailed)	<0.01	<0.01
	N	10	10

** Correlation is significant at the 0.01 level (2-tailed)

Rasprava

Protetski nadomjestak pacijenti bi trebali u cijelosti prihvatiti jer im na dulje razdoblje nadoknađuje funkciju i estetiku (34 – 36). Cirkonij-oksidna keramika danas je jedan od najčešće korištenih sustava potpune keramike, kako za sanaciju pojedinačnim krunicama tako i u mosnim konstrukcijama. U kliničkom radu uočilo se da nastaju gubici funkcijske trajnosti nadomjestka zbog dvaju problema – delaminacije i *chippinga* dijela obložne keramike. Kako bi se otkrio uzrok (8, 9) i pokušao eliminirati taj problem, površina cirkonij-oksidne jezgre tretirala se na različite načine (19 – 23). Pjeskarenje je jedan od mogućih načina obrade kako bi se postigla snažna veza s obložnom keramikom. No, naprezanje izazvano tim postupkom inicira transformaciju zrna cirkonijeva oksida na površini u monoklinsku fazu koja je volumenom veća te rezultira tlačnim naprezanjem i smanjenjem koeficijenta termičke istezljivosti (23, 24, 37, 38). Iz tog razloga transformirana se zrna trebaju vratiti u tetragonalnu fazu postupkom koji se naziva regeneracijsko pečenje, čime se usklađuje KTI (24).

Vezna čvrstoća je ključno naprezanje koje materijal može podnijeti do loma kad ga se izloži smičnom opterećenju. Osobito je važna kod svih istraživanja koja proučavaju spojeve dvaju materijala kod višeslojnih sustava (39).

U ovom istraživanju procjenjivale su se promjene morfologije i hrapavosti površine cirkonijeva oksida. Pjeskarenje česticama aluminijeva oksida veličine 50 i 125 µm provedeno je referirajući se na prijašnje studije (22, 25, 26, 40). Nakamura i suradnici (41) zabilježili su povećanje vezne čvrstoće između Y-TZP-a i obložne keramike zahvaljujući povećanju hrapavosti površine uzrokovanu pjeskarenjem. No, druga studija pokazala je smanjenje vezne čvrstoće pjeskarenjem (42). Iako neke studije upućuju na to da se vezna čvrstoća povećava povećanjem hrapavosti površine, u drugima se, pak, ističe da transformacija zrna na površini utječe na smanjenje vezne čvrstoće (25). Kad je riječ o problemu vezne čvrstoće, još se vode mnogobrojne polemike, pa se ovim istraživanjem željela ustanoviti povezanost hrapavosti površine i vezne čvrstoće obložne keramike. Dokazano je da hrapavost površine značajno utječe na veznu čvrstoću. Nakon testa vezne čvrstoće, rezultati analize loma pokazali su da je u drugoj skupini uzoraka (polirani i pjeskareni) obložna keramika ostala na površini cirkonijeva oksida nakon znatno većih smičnih sila. Može se zaključiti da je adhezivna čvrstoća obložne keramike veća od kohezivne. Najslabija karika nije spoj cirkonijeva oksida i obložne keramike, nego sama obložna keramika. Pjeskarenje je nahrapavilo, te time pove-

Discussion

Prosthodontic appliance must be fully accepted by the patient to increase his/her quality of life, but it also has to show long term durability and clinical quality (34-36). Zirconia is one of the most frequently used all-ceramic systems for dental crowns and FPDs. However, during clinical use, it has been observed that delamination and chipping were the most frequent reasons for failure. To discover the cause of failure (8, 9), the surface of zirconia core has been variously pre-treated (19-23). Air-abrasion of the zirconia surface is an important treatment method in achieving strong bond to veneering porcelain. However, the mechanical stress initiates a phase transition to monoclinic zirconia, and the phase transition gives rise to volume expansion resulting in compressive stress and reduction in coefficient of thermal expansion (23, 24, 37, 38). Therefore, the phase transition needs to be reversed to the tetragonal zirconia by recommended heat treatment prior to veneering also known as regeneration firing, with a concomitant alteration in the coefficient of thermal expansion (24).

Shear strength is the maximum stress that a material can withstand before failure in a shear mode of loading and is particularly valuable in the study of interfaces between materials (39). This study evaluated the surface morphology and roughness of Y-TZP ceramic material after grinding as well as grinding and sandblasting in relation to shear bond strength of the bi-layered veneering ceramics. Sandblasting with 50 and 125 µm alumina was performed with reference to earlier studies (22, 25, 26, 40). Nakamura et al. (41) reported that bond strength between TZP and veneering ceramics increased due to a rise in the surface roughness of the TZP by sandblasting. However, another study reported that bond strength decreased (42). Although some studies have indicated that bond strength increases on surfaces with increased roughness, others have suggested that phase transformation of TZP by sandblasting may adversely affect bond strength (25). There is still a lot of controversy concerning this problem, so we aimed to find a correlation between surface roughness of zirconia samples and shear bond strength values of veneered ceramics. In the present study, surface roughness showed significant influence on the bond strength. After the shear test, the results of the fracture analysis showed that in the second group (ground and sandblasted samples) the veneering ceramic remained on the zirconia surface under significantly higher shear forces. It could be concluded that the adhesive strength between zirconia and the veneering ceramic was higher than the cohesive strength of the veneering ceramćalo i preoblikovalo, veznu površinu cirkonij-oksidne keramike. Kim i suradnici proveli su *in vitro* studiju čiji su rezultati pokazali da pjeskarenje znatno povećava veznu čvrstoću s obložnom keramikom, za razliku od aplikacije veznog sredstva (32). No, rezultati Fischera i njegovih kolega pokazuju da se pjeskarenje i regeneracijsko pečenje ne preporučuju kao načini obrade površine (4).

Rezultati dobiveni u ovom istraživanju upućuju na snažnu i značajnu povezanost parametara hrapavosti površine i vrijednosti vezne čvrstoće te se ističu potreba za daljnjim istraživanjima na većem broju uzoraka.

Zaključak

Ustanovljena je međusobna povezanost hrapavosti površine cirkonijeva oksida i vezne čvrstoće s obložnom keramikom te će se daljnja istraživanja usmjeriti prema rasvjetljavanju problema povezanosti navedenih parametara na većem broju uzoraka.

Zahvala

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Sukob interesa

Nije bilo sukoba interesa.

ic. The weakest link was not the interface but the veneering ceramic itself. Air-abrasion roughened the zirconia, increased the bonding area and modified the ceramic surface. Kim et al. concluded that the mean *in vitro* shear bond strength of veneering ceramic on zirconia treated with airborne-particle abrasion was significantly higher than that subjected to liner-applied treatments (32). However, the results obtained by Fischer et al. showed that sandblasting was not a necessary surface pretreatment to enhance bond strength and that regeneration firing was not recommended (4).

Preliminary results obtained in the present study show strong, positive and significant correlation between surface roughness parameters and shear bond strength values (SBS) and point out the need for further research between different surface roughness parameters and SBS values of veneering ceramics on a larger sample.

Conclusion

The positive relation between shear bond strength values and surface roughness has been proved and further research will be directed towards the relation between the two parameters on a larger sample.

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Conflict of interest

No conflict of interest.

Abstract
Delamination and chipping of veneering porcelain were described as the most frequent reasons for the failure of zirconia fixed partial dentures. **Aim:** The aim of the study was to assess the correlation of surface roughness and the shear bond strength of veneering ceramics to zirconia core. **Materials and methods:** A total number of 10 zirconia samples, 5 in each group were sintered. Samples in the first group were ground, and in the second were ground and sandblasted. After the surface treatment, veneering ceramic was fired to the core up to the thickness of 3 mm, the samples were embedded in the PTFE molds using PMMA resin and subjected to shear force until fracture occurred. The surface roughness was assessed prior to application of veneering ceramics by the Stylus instrument Perthometer S8P and Ra (mean roughness) and Z values (distance from the highest to the lowest point of measurement along the observed line) were obtained. **Results:** Ground and sandblasted samples had significantly rougher surface ($p<0.01$) and showed significantly higher shear bond strength ($p<0.01$). **Conclusion:** The positive and significant correlation was found between surface roughness values and shear bond strength. Further research is needed to completely elucidate the observed relation.

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Key words
Dental Veneers; Zirconium oxide; Ceramics; Surface Properties; Shear Strength; Dental Cements

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