Anna M. RYNIEWICZ*, Andrzej RYNIEWICZ**, Łukasz BOJKO***, Wojciech RYNIEWICZ****

ANALYSIS OF THE IMPACT OF BIOMATERIAL AND THE TECHNOLOGY OF PROSTHETIC CROWNS MANUFACTURING ON THE CONNECTION WITH VENEERING CERAMICS

ANALIZA WPŁYWU BIOMATERIAŁU I TECHNOLOGII WYTWORZENIA KORON PROTETYCZNYCH NA ŁĄCZENIE Z CERAMIKĄ LICUJĄCĄ

Kev words:	layered structures. CAD/CAM milling. SLM, concentrated contact, microhardness.				
Abstract:	The aim of the study is to identify the endurance parameters of prosthetic crowns veneered with dedi ceramics on metal, glass-ceramic, and ceramic frameworks. Metal frameworks were made using CAD/0 milling technology and SLM technology, while the glass-ceramic and ceramic frameworks were proc using only the CAD/CAM milling technology. The research materials are samples replicating the lay structures of prosthetic crowns. The veneering procedure must ensure the adhesion of the ceramics to the bearing framework. The tests modelling the conditions of concentrated loads during chewing were carrie using the Instron 3345 testing machine. Determination of microhardness in cross-sections through lay structures of crowns was performed using the HMV Micro Hardness Tester. The comparison of force los the indenter as a function of penetration depth indicates that the value of the maximum depth depen- the configuration of microhardness of the framework and dentine. The zirconium ceramics ZrO_2 (3Y- – veneered with Elephant Sakura silica ceramics – should be indicated as the most advantageous ma composition.				
Słowa kluczowe:	struktury warstwowe, frezowanie CAD/CAM, SLM, styk skoncentrowany, mikrotwardość.				
Streszczenie:	Celem pracy jest identyfikacja parametrów wytrzymałościowych koron protetycznych licowanych dedyko- wanymi ceramikami na podbudowach metalowych, szklanoceramicznych i ceramicznych. Podbudowy me- talowe zostały wytworzone w technologii frezowania CAD/CAM i w technologii SLM, a podbudowy szkla- noceramiczne i ceramiczne w technologii frezowania CAD/CAM. Materiałem badań są próbki replikujące warstwowe struktury koron protetycznych. W wyniku procedury licowania musi być zapewniona adhezja ceramiki do podbudowy nośnej. Badania modelujące warunki skoncentrowanych obciążeń podczas żucia wykonano na maszynie wytrzymałościowej Instron 3345. Wyznaczenie mikrotwardości w przekrojach przez warstwowe struktury koron zrealizowano na maszynie HMV Micro Hardness Tester. Zestawienie siły obcią- żającej wgłębnik w funkcji głębokości penetracji wskazuje, że wartość maksymalnego zagłębienia, zależy od konfiguracji mikrotwardości podbudowy i mikrotwardości dentyny. Jako najkorzystniejszą kompozycję materiałową należałoby wskazać ceramikę cyrkonową ZrO ₂ (3Y – TZP) – licowaną ceramiką krzemionkową Elephant Sakura.				

ORCID: 0000-0003-2469-6527. Jagiellonian University Medical College, Faculty of Medicine, Dental Institute, Department of Dental Prosthodontics, Montelupich 4 Street, 31-155 Cracow, Poland.

^{**} ORCID: 0000-0003-3437-7650. State University of Applied Science, Institute of Technology, Zamenhofa 1a Street, 33-300 Nowy Sącz, Poland.

^{***} ORCID: 0000-0002-6024-458X. AGH University of Science and Technology, Faculty of Mechanical Engineering and Robotics, Mickiewicza 30 Ave., 30-059 Cracow, Poland.

^{****} ORCID: 0000-0002-9140-198X. Jagiellonian University Medical College, Faculty of Medicine, Dental Institute, Department of Dental Prosthodontics, Montelupich 4 Street, 31-155 Cracow, Poland.

INTRODUCTION

Ceramics are materials which best reproduce the natural tooth tissues in terms of aesthetics [L. 1–5]. They are used for the production of ceramic-metal crowns and bridges, as well as for manufacturing all-ceramic restorations [L.6]. However, ceramic is brittle and sensitive to tension. while the endurance and aesthetics of the prosthetic restoration depends on the biomaterial and production technology. Correct design of the restoration is essential. Clinical preparation of the teeth should spare the hard tissues as much as possible and provide space for the appropriate thickness of the load-bearing framework, as well as for the optimal thickness of the ceramics, which determines the aesthetics of the restoration. In some situations, ceramic-metal restorations have an advantage over all-ceramic restorations. The loadbearing framework must be of sufficient stiffness and strength to protect the abutment teeth, reduce deflections and deformations of the structure, and decrease stress concentrations in the veneering ceramics and in the connection zone of the load-bearing structure and the veneering layer [L. 7-9]. In the case of ceramic-metal structures, thermal expansion coefficients of ceramics and metal must be compatible so that the ceramics do not crack during the production of a restoration. The system should be designed so that the values of the expansion are slightly higher for the metal than for the ceramics and cause its compression when cooling after firing. The veneering layers of the framework are responsible for the functional and biomechanical cooperation with the opposing teeth [L. 10, 11]. They should be able to withstand occlusive and chewing loads. The lost-wax casting technology is a traditional method of making a load-bearing structure for prosthetics. In recent years, the CAD/CAM system has been introduced which uses scanning and virtual mapping of the prosthetic base, as well as numerical design of the restoration and computer-aided production of the load-bearing structure using milling from factory matrices or laser sintering of metal powders [L. 12-14]. The load-bearing structure is subjected to successive stages of veneering with the appropriate set of ceramics.

The aim of the study is to determine endurance parameters of layered structures for prosthetic crowns veneered with dedicated ceramics on metal, glassceramic, and ceramic frameworks. Metal frameworks were made using CAD/CAM milling technology and SLM technology, while the glass-ceramic and ceramic frameworks were produced using only the CAD/CAM milling technology.

MATERIAL AND METHOD

The research materials were specimens replicating the layered structure of prosthetic crowns on frameworks made using digital technologies. The quality of the ceramic veneering layer depends on biomaterial of the framework and on the subsequent stages of applying and firing the ceramic opaque, dentin, and enamel layers. The veneering procedure must ensure the adhesion of the ceramics to the framework, which can be made of metal or ceramics [L. 15–17].

The following structures were tested:

- Ti6Al4V and TiCP frameworks milled using CAD/ CAM and Ti6Al4V frameworks sintered using SLM – veneered with Vita Titankeramik ceramics,
- CoCrMo frameworks milled using CAD/CAM and SLM sintering technology – veneered with the Duceram Kiss set,
- Li₂Si₂O₅ glass-ceramic frameworks milled using CAD/CAM – veneered with IPS e.max Ceram ceramics, and
- ZrO₂ (3Y-TZP) ceramic frameworks milled using CAD/CAM and sinterized veneered with Elephant Sakura ceramics.

The sets of test samples were made in professional laboratories in accordance with the latest knowledge about innovative technologies of prosthetic structures [L. 2-4, 9, 18]. Samples of materials for load-bearing frameworks in the form of $\phi 1/4$ "discs with a thickness of 1/16", in sets of 9 pieces, were made from factory milling matrices using CAD/CAM from Ti6Al4V alloy, TiCP titanium, CoCrMo alloy and Li₂Si₂O₅ ceramics as well as ZrO, ceramics - milling technology using the CORiTEC 350i device by imes icore. The ZrO, discs were milled adequately larger (expansion coefficient of 24%) so that after sintering they achieved the proper structure and dimensions. The discs of materials for load-bearing frameworks made using the Selective Laser Melting (SLM) method in the CAD/CAM system, 9 pieces each, were sintered from CoCrMo powders and Ti6Al4V powders using a Renishaw AM250 machine. The samples prepared in this way, i.e. produced using the milling technology and the incremental sintering technology, were veneered with successive layers of dedicated ceramics.

Samples, replicating layered structures of prosthetic crowns, in the form of discs (3 pieces of each type) were subjected to endurance tests performed using an Instron 3345 machine in concentrated contact which modelled the loads obtained during chewing (**Fig. 1a**). The discs were mounted on a test table. The following test parameters were used: the maximum load of the indenter of 20 N, the speed of loading and unloading of 40 N/min, and the time of maintaining the maximum force -5 s. During the test, the depth of penetration into the disc structure was continuously recorded.

After having previously made the specimens, the remaining sets (3 discs from each structure) were subjected to microhardness tests using the Vickers method and the HMV Micro Hardness Tester by Shimadzu (Fig. 1b). HV0.1 microhardness tests were performed, with the nominal value of the loading force of 980.7 mN.

The preparation of the samples consisted of sinking the discs in resin. After the resin condensation, the discs were mounted in a special holder and cut with a diamond disc in a water bath. The next stage of sample preparation was multi-stage polishing using an automatic polisher by Struers and degreasing the surface of the specimens (**Fig. 2**).



Fig. 1. Test equipment: a) Instron 3345 testing machine, b) HMV Micro Hardness Tester

Rys. 1. Urządzenia badawcze: a) maszyna wytrzymałościowa Instron 3345, b) twardościomierz HMV Micro Hardness Tester



Fig. 2. Specimens for microhardness testing by the Vickers method Rys. 2. Zgłady do badań mikrotwardości metodą Vickersa

RESULTS AND DISCUSSION

In titanium samples milled using CAD/CAM and in samples sintered using SLM – veneered with ultralow-melting point Vita Titankeramik ceramics, the values of the maximum depth of penetration slightly differed (**Fig. 3**). The greatest depth was reported in the TiCP framework, which had the lowest microhardness (Tab. 1, Fig. 7). With significant differences in microhardness of titanium frameworks, milled TiCP – 174.60 HV, Ti6Al4V from SLM – 328.00 HV, Ti6Al4V milled – 378.83 HV, there were no significant differences in microhardness, neither in the opaque nor in the dentine layers. The highest values of microhardness in opaque and dentin ceramics were found for samples from the Ti6Al4V framework made using the SLM technology.

In CoCrMo samples milled using CAD/CAM and in CoCrMo samples sintered using SLM – veneered with high-melting point Duceram Kiss ceramics, the values of the maximum depth of penetration were very similar (**Fig. 4**). A larger depth was observed in milled CoCrMo frameworks, and a slightly smaller depth was observed in sintered frameworks.

In CoCrMo frameworks, there were significant differences in microhardness after veneering, depending on the manufacturing technology. The average value of the microhardness of the framework made using the SLM was 644.80 HV, and using the CAD/CAM milling technology of sintered powder discs, it was almost half lower and amounted to 328.20 HV (Tab. 2, Fig. 8). The results of these tests confirmed the outcomes of the previously performed experiments, which showed that the CoCrMo alloy obtained using the DMLS sintering (before the veneering process) had a microhardness of 6582.3 MPa, and using the milling of sintered powder discs - much lower i.e. 4951.0 MPa [L. 13]. It should be clarified that the test results obtained with the Oliver's and Pharr's method are expressed in MPa, while, in the Vickers method, the results are given in HV. There is no unequivocal formula for converting units, and it can only be approximated that the value of microhardness in HV is approximately 10 times lower than that obtained in the Oliver's and Pharr's method. Analysing the results

of microhardness before and after the veneering process, we can conclude that high temperatures of the veneering process, especially with high-melting point ceramics, may reduce the value of microhardness in CoCrMo alloys due to structural changes.



- Fig. 3. The relationship between the force loading the indenter as a function of the depth of penetration into the structures of prosthetic crowns made on titanium frameworks veneered with Vita Titankeramik ceramics
- Rys. 3. Zależność siły obciążającej wgłębnik w funkcji głębokości penetracji w struktury koron protetycznych na podbudowach tytanowych licowanych ceramiką Vita Titankeramik
- Table 1. Microhardness of material compositions veneered with the Vita Titankeramik ceramics made on titanium frameworks
- Tabela 1. Mikrotwardości w kompozycjach materiałowych licowanych zestawem Vita Titankeramik na podbudowach tytanowych

	Statistical parameter	Material composition					
Measurement layer		Vita Titankeramik + Ti6Al4V with milling technology		Vita Titankeramik + Ti6Al4V with SLM technology		Vita Titankeramik + TiCP with milling technology	
		Microhardness, HV	The average diameter of indent, μm	Microhardness, HV	The average diameter of indent, μm	Microhardness, HV	The average diameter of indent, μm
Veneering layer	Average	602.25	17.76	640.75	17.03	578.50	18.00
	Standard deviation	123.29	2.36	36.73	0.66	76.24	1.34
Opaque layer	Average	594.67	17.80	604.750	17.62	561.60	18.25
	Standard deviation	92.20	1.83	89.794	1.43	69.13	1.29
Framework	Average	378.83	22.17	328.00	23.83	174.6	32.60
	Standard deviation	30.60	1.00	26.97	0.96	9.40	1.48



- Fig. 4. The relationship between the force loading the indenter as a function of the depth of penetration into the structures of prosthetic crowns made on CoCrMo frameworks veneered with Duceram Kiss ceramics
- Rys. 4. Zależności siły obciążającej wgłębnik w funkcji głębokości penetracji w struktury koron protetycznych na podbudowach CoCrMo licowanych ceramiką Duceram Kiss

In material compositions made on CoCrMo frameworks, very high and similar microhardness of oxide structures in the opaque zone is characteristic, in the range of 813.80 HV–807.40 HV, regardless of the differences in microhardness of the frameworks. Lower values of microhardness occur in the dentine layers. The microhardness of the dentine layer is slightly higher for the milled framework than for the structures made on the SLM framework.

The compositions made on metal frameworks are characterized by high values of microhardness in the opaque layers for much harder CoCrMo frameworks than for titanium frameworks.

In samples from milled glass-ceramic frameworks veneered with glass nanofluoroapatite ceramics, the maximum depth of penetration into the structure of 27.23 μ m, which was similar to the depth obtained in titanium samples veneered with ultra-low-melting point Vita Titankeramik ceramics (**Fig. 5**).

	Statistical parameter	Material composition					
Measurement layer		Duceram k with milli	Kiss + CoCrMo ng technology	Duceram Kiss + CoCrMo with SLM technology			
		Microhardness, HV	The average diameter of indent, μm	Microhardness, HV	The average diameter of indent, μm		
Veneering layer	Average	643.40	16.98	644.50	19.96		
	Standard deviation	15.61	0.55	20.68	0.34		
Opaque layer	Average	813.80	15.13	856.00	14.77		
	Standard deviation	73.19	0.88	92.11	1.27		
Framework	Average	328.2	23.82	644.8	16.96		
	Standard deviation	27.50	1.05	23.47	0.32		

 Table 2.
 Microhardness of material compositions veneered with Duceram Kiss ceramics made on cobalt frameworks

 Tabela 2.
 Mikrotwardości w kompozycjach materiałowych licowanych zestawem Duceram Kiss na podbudowach kobaltowych

The greatest maximum depth of 37.60 μ m was found for samples from milled and sinterized zirconium oxide ZrO₂ (3Y–TZP) frameworks veneered with Elephant Sakura silica ceramics (**Fig. 5**).

Glass-ceramic and ceramic frameworks are characterized by high microhardness (**Tab. 3**, **Fig. 8**). The ceramic ZrO₂ (3Y–TZP) framework has a microhardness of 1415.80 HV. It can be concluded

that such a configuration of a very high value of microhardness in the zirconium framework and a much softer dentine layer (603.60 HV) determine the greatest depth of penetration into the structure in the test of concentrated load. At the same time, it should be added that this material composition does not contain an opaque layer, since it is unnecessary to remove the metal colour.



- Fig. 5. The relationship between the force loading the indenter as a function of the depth of penetration into the structures of prosthetic crowns made on glass-ceramic frameworks veneered with IPS e.max Ceram ceramics and on ceramic frameworks veneered with Elephant Sakura ceramics
- Rys. 5. Zależności siły obciążającej wgłębnik w funkcji głębokości penetracji w struktury koron protetycznych na podbudowach szklanoceramicznych licowanych ceramiką IPS e.max Ceram i ceramicznych licowanych ceramiką Elephant Sakura

The summary of the relationships between the force loading the indenter as a function of the penetration depth indicates that the maximum depth may depend on the configuration of the microhardness of the framework and the dentine layer (**Tables 1–3, Fig. 6**).

A low value of penetration depth is observed for slightly differentiated microhardness values, an example of which is the $\text{Li}_2\text{Si}_2\text{O}_5$ framework, veneered with IPS e.max Ceram glass ceramics (**Fig. 9**).

Referring the values of penetrator depth to the situation of concentrated loads during chewing, it should be mentioned that flexible and at the same time non-chipping veneering is the optimal biomechanical solution for the teeth in contact [L. 5, 19, 20].

The tests performed in cross-sections through material compositions of crowns made on titanium, cobalt-chrome, glass-ceramic, and ceramic frameworks have shown that the average microhardness of frameworks has significantly different values (**Fig. 10**) as follows:

• The lowest microhardness is characteristic of titanium frameworks, in the range of 578.50–602.25 HV, the

Table 3. Microhardness of material compositions veneered with IPS e.max Ceram made on Li₂Si₂O₅ frameworks and with Elephant Sakura ceramics on ZrO, frameworks

Tabela 3. Mikrotwardości w kompozycjach materiałowych licowanych ceramiką IPS e.max Ceram na podbudowach Li₂Si₂O₅ oraz ceramiką Elephant Sakura na podbudowach ZrO₂

	Statistical parameter	Material composition				
Measurement layer		IPS e.max Co with millin	eram + Li ₂ Si ₂ O ₅ ng technology	Elephant Sakura + ZrO ₂ with milling technology		
		Microhardness, HV	The average diameter of indent, μm	Microhardness, HV	The average diameter of indent, μm	
Veneering layer	Average	672.40	16.64	603.60	17.54	
	Standard deviation	53.43	0.72	31.15	0.53	
Opaque layer	Average	643.70	16.99	_	_	
	Standard deviation	35.64	0.62		_	
Framework	Average	663.27	16.74	1415.8	11.46	
	Standard deviation	39.06	0.62	103.42	0.44	

lowest value was found for the TiCP framework obtained using milling, and the highest value was noted for Ti6Al4V also from the milling technology.

- Even higher values than for cobalt-chrome frameworks, have been observed for milled glass-ceramic frameworks 672.40 HV,
- The group of cobalt-chrome frameworks has higher microhardness, in the range of 643.40–644.50 HV, while the framework obtained by sintering has a higher microhardness.
- Several, up to 2.5 times, higher values of microhardness were noted for ZrO₂ (3Y–TZP) frameworks.



Fig. 6. The relationship between the force loading the indenter as a function of the depth of penetration into the layered structures of prosthetic crowns

Rys. 6. Zależność siły obciążającej wgłębnik w funkcji głębokości penetracji w warstwowe struktury koron protetycznych



- Fig. 7. A list of average values of microhardness of layered compositions of titanium biomaterials building prosthetic crowns veneered with Vita **Titankeramik ceramics**
- wartości mikrotwardości Rys. 7. Zestawienie średnich w kompozycjach warstwowych biomateriałów tytanowych budujących korony protetyczne licowanych ceramiką Vita Titankeramik

Dentin layers in the tested compositions have more uniform microhardness. The highest value of microhardness (672.40 HV) of the veneering layer was found for the glass-ceramic Li₂Si₂O₅ framework, veneered with IPS e.max Ceram glass ceramics. This composition showed the lowest susceptibility to indentation, with the value of 27.23 µm, which may result from the high microhardness of the veneering laver and cause unfavourable contact with the opposing teeth with the natural enamel.

The zirconium ceramics ZrO₂ (3Y-TZP) – veneered with Elephant Sakura silica ceramics should be indicated as the most advantageous material composition in



- Fig. 8. A list of average values of microhardness of layered compositions of cobalt biomaterials building prosthetic crowns veneered with Duceram Kiss ceramics

Rys. 8. Zestawienie średnich wartości mikrotwardości w kompozycjach warstwowych biomateriałów kobaltowych budujących korony protetyczne licowanych ceramika Duceram Kiss



- Fig. 9. A list of average values of microhardness of the layered compositions of Li₂Si₂O₅ biomaterials veneered with IPS e.max Ceram and ZrO, (3Y-TZP) biomaterials veneered with Elephant Sakura ceramics
- Rys. 9. Zestawienie średnich wartości mikrotwardości kompozycjach warstwowych biomateriałów w Li₂Si₂O₅ licowanych ceramiką IPS e.max Ceram oraz biomateriałów ZrO₂ (3Y-TZP) licowanych ceramiką Elephant Sakura

terms of biomechanical properties, biocompatibility, endurance, and aesthetics.

In the group of titanium frameworks, the best parameters were obtained for the Ti6Al4V composition made using the SLM technology, veneered with



 Fig. 10. A list of average values of microhardness of the layered structures of biomaterials building prosthetic crowns
 Rys. 10. Zestawienie średnich wartości mikrotwardości w strukturach warstwowych biomateriałów budujących korony protetyczne

Vita Titankeramik ceramics, and in cobalt-chrome frameworks, which was the CoCrMo framework made using the SLM technology, veneered with Duceram Kiss ceramics.

CONCLUSIONS

The comparison of the force loading the indenter as a function of the penetration depth indicates that the maximum penetration depth depends on configuration of the microhardness of the framework and dentine.

In all the compositions, no chipping of the veneering layer of ceramics was found under concentrated indenter loading. The highest value of microhardness of the veneering layer was found for the glass-ceramic $\text{Li}_2\text{Si}_2\text{O}_5$ framework, veneered with IPS e.max Ceram glass ceramics, which may cause that the crown contacting the natural opposing teeth causes damage of their enamel.

The zirconium ceramics ZrO_2 (3Y-TZP) – veneered with Elephant Sakura silica ceramics – should be indicated as the most advantageous material composition in terms of biomechanical properties, biocompatibility, endurance, and aesthetics.

ACKNOWLEDGEMENT

This work is financed by AGH University of Science and Technology, Faculty of Mechanical Engineering and Robotics: subvention No. 16.16.130.942.

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