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STUDY OF THE IMPACT OF INCREMENTAL TECHNOLOGY ON MECHANICAL AND TRIBOLOGICAL PROPERTIES OF BIOMATERIALS

BADANIE WPŁYWU TECHNOLOGII PRZYROSTOWEJ NA WŁAŚCIWOŚCI MECHANICZNE BIOMATERIAŁÓW

Key words: biomaterials, technology, nanoindentation, SEM, friction, wear, biomechanical estimation.

Abstract The latest method for creating denture replenishment in CAD / CAM systems is Direct Metal Laser Sintering (DMLS) technology. With the use of DMLS, prosthodontics, implant prosthetics, and facial-cranial-jaw surgery adapted to individual patient conditions can be realized. The aim is to evaluate the strength, microstructure, and tribological properties of Ti6Al4V and CoCrMo alloys obtained from DMLS technology in the aspect of therapeutic constructions. The conducted tests show that, in the DMLS technology, as compared to milling technology preceded by casting and forging or pressed powder and sintering, for the same percentage composition of elements, the micromechanical properties, microstructural and tribological change. This procedure, from which constructions for various dental applications are obtained, is the new technology preferred for making permanent restorations faced with ceramics, producing intravascular implants, and implants of the temporomandibular joint. It can be an alternative to conventional cast-based methods and CAD / CAM based milling.

Słowa kluczowe: biomateriały, technologia, nanoindentacja, SEM, tarcie, zużycie, ocena biomechaniczna.

Streszczenie Najnowszą metodą tworzenia uzupełnień stomatologicznych w systemie CAD/CAM jest technologia Direct Metal Laser Sintering (DMLS). Z wykorzystaniem DMLS można realizować wykonanie konstrukcji protetyki, implantoprotetyki oraz chirurgii twarzowo-czaszkowo-szczękowej dostosowanej do indywidualnych warunków pacjenta. Celem jest ocena parametrów wytrzymałościowych, mikrostruktury i właściwości tribologicznych stopów Ti6Al4V oraz CoCrMo uzyskanych w technologii DMLS w aspekcie zastosowania na konstrukcje terapeutyczne. Przeprowadzone badania wskazują, że w technologii DMLS, w porównaniu z technologią frezowania poprzedzoną procesami odlewania i kucia lub prasowania proszku i spiekania, dla tego samego składu procentowego pierwiastków, zmieniają się właściwości mikromechaniczne, mikrostrukturalne i tribologiczne. Procedura ta, w wyniku której uzyskuje się konstrukcje dla różnych aplikacji stomatologicznych – jest nową technologią preferowaną do wykonawstwa uzupełnień stałych licowanych ceramiką, do wytwarzania wszczepów śródkostnych oraz implantów stawu skroniowo-żuchwowego. Może ona stanowić alternatywę dla klasycznych metod opartych na odlewnictwie oraz metod CAD/CAM opartych na frezowaniu.

INTRODUCTION

The application of latest manufacturing technologies of dental constructions aims at using such materials that meet the highest requirements in the range of biocompability, strength, and aesthetic as well as reaching accuracy in shape mapping. The traditional technology is the lost-wax method, and its disadvantages are casting shrinkage, imperfections in the microstructure of material and laborious stages in the realization of the construction **[L. 1–5]**.

The CAD/CAM system is used in the contemporary stomatology for computer aided design (CAD) of the construction as well as for their manufacture in

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a computer-controlled process on the basis of created numerical model (CAM). The CAM procedure allows creating constructions with the help of the milling technology from factory fittings; however, the most current method is Direct Metal Laser Sintering (DMLS) technology **[L. 6–14]**. Aided by the DMLS, one can build constructions for prosthetics, implantology, and facial-jaw surgery, adapted to individual patient conditions. Designs prepared in the CAD procedure are produced in the CAM stage during the sintering process with a laser beam on selective metal powders in the successive layers.

The aim of the work is to evaluate the strength parameters, microstructure, and tribological properties of biomaterials obtained from DMLS technology, in terms of the use of therapeutic construction for stomatology.

MATERIAL AND METHODS

The material for tests were samples from Ti6Al4V alloy and from CoCrMo alloy produced from selective powders with the help of the incremental technology in CAD/CAM **(Fig. 1)**. The samples from the same alloy elements obtained through milling in CAD/CAM **(Fig. 2)** were tested as reference materials.

In the milling process, the factory discs were made from the following:

- Ti6Al4V alloy after casting and forging, Starbond Ti5 Disc (Grade 5) ELI Ti6Al4V according to the ISO 5832-3:2016, and
- CoCrMo alloy after the sintering process of the pressed powder, Starbond CoS Soft Disc, type 4 according to the EN ISO 22674.

The samples sets were taken from each material. The first set of samples was prepared in such a way that the surface layer intended for tests was created in the plane of the moving laser beam. The second set included perpendicular cross-sections. Titanium alloy Ti6Al4V is designed for restorations embedded on implants, superstructures, fasteners, beams as well as crowns and bridges. CoCrMo from the sintering process based on binder and pressed powder is for the manufacturing of permanent prosthetic restorations: milled crowns, bridges, beams, implant surges and connectors.

Micromechanical and microstructural tests were carried out on properly prepared metallographic specimens. For this purpose, the samples were embedded in a resin and subjected to polishing on the Struers TegraForce-5, in which, during the programmed operations, the surface layer of the samples required for tests was reached.

The samples to tribological tests had a disc shape with a diameter 6.35 mm and a thickness of 2 mm **(Fig. 3)**. The samples were made from powders EOS Titanium Ti64 and EOS CobaltChrome SP2 with the incremental technology. In the device dedicated to dental works, the powder layer with the thickness of 0.02 mm is applied on the working platform. After that, the laser beam in the infrared range is conducted upon the surface of the powder according to the bitmap being a virtual record of the created substructure. Then, the working platform lowers and another layer of powder is applied. Rapid melting curing results in a homogeneous material structure. Reference discs were obtained from the CAD/CAM milling method from cast profile on the 5-axis dental milling machine CORiTEC 350i imes-icore.

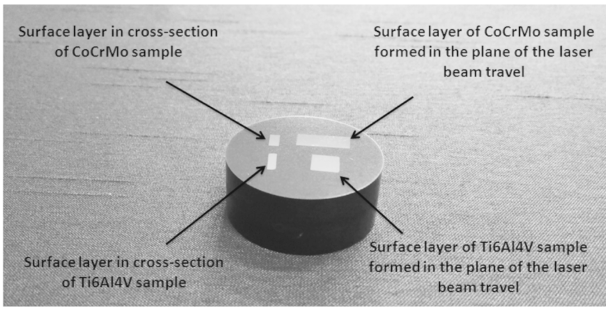

Fig. 1. Ti6Al4V and CoCrMo samples from DMLS for micromechanical and microstructural tests
Rys. 1. Próbki Ti6Al4V oraz CoCrMo z technologii DMLS do badań mikromechanicznych i mikrostrukturalnych

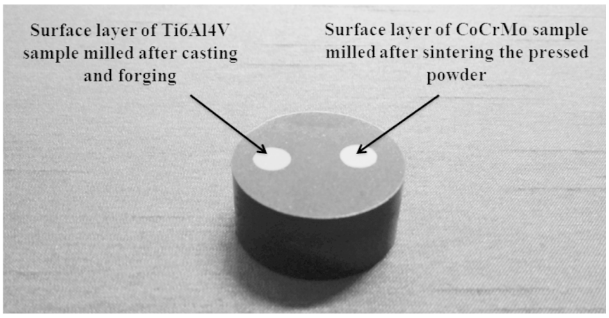

Fig. 2. Ti6Al4V and CoCrMo samples milled in CAD/CAM from factory discs for micromechanical and microstructural testing
Rys. 2. Próbki Ti6Al4V oraz CoCrMo frezowane w CAD/CAM z fabrycznych dysków do badań mikromechanicznych i mikrostrukturalnych

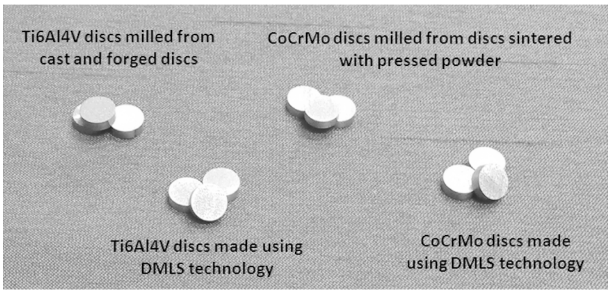

Fig. 3. Samples for tribological tests
Rys. 3. Próbki do badań tribologicznych

The micromechanical properties tests, which included microhardness and Young's modulus measurement, were performed on Micro Combi Tester CSEM Instruments. The parameters were set on the basis of the indentation of the sample aided by the pyramid shaped proper diamond penetrator **[L. 15]**. Strength values and penetration depth of blade were continuously recorded in a load and unload cycle during the whole test. The maximum load was 100 mN, the loading and unloading speed was 200 mN/min, and the time to maintain the maximum load – 5 s. The dependencies of indenter load as a function of penetration depth were designated for each cycle. Analysis of micromechanical properties was based on the Oliver's and Pharr's method by which the microhardness (HV) and elasticity modulus (E) were designated from the penetration curve.

Microstructural tests of titanium Ti6Al4V alloys and cobalt CoCrMo alloys were carried out with a Hitachi S3400N scanning microscope with EDS and EBSD attachments.

The tests of the coefficient of friction and wear resistance of tested samples prepared in two different technological processes were carried out in a ball-three discs (from tested materials) system in physiological saline using the Four Ball Wear Tester Brown by Roxana Machine Works. Counter samples were balls of ø ½" diameter according to the PN-83/M-86452. The test parameters were as follows:

- Rotational speed 200 Rpm ± 50 Rpm,
- Working temperature 36.6°C ± 1.5°C,
- Load 100 N ± 10N, and
- Duration 15 min ± 10 s.

The measure of anti-wear properties of the tested materials was the average value of the wear defect on the discs at constant test parameters. The measurements of the diameter of wear defects were performed perpendicular and parallel to the friction traces of the Hitachi S3400N high-resolution scanning microscope with EDS and EBSD attachments. During the tests, the coefficient of friction was continuously recorded. The wear resistance values were characterized by descriptive statistics: mean value, standard deviation, and scatter scores. Tukey's test helped to compare the parameters. It is referred to as an intermediate test between Newman-Keuls and Scheffe tests. Values of the statistics were tested at a significance level of $p < 0.05$. Calculations were made with the Statistica 13.1 package.

RESULTS AND DISCUSSION

Results of micromechanical tests, micro Hardness and Young's Modulus – the MCT, were measured as a mean of 8 measurements for each sample. **Tables 1** and **2** summarize the results of micromechanical studies by Oliver and Pharr for Ti6Al4V alloy and CoCrMo alloy. The maximum penetration depth hmax, hardness H, and elasticity modulus E at 100 mN perimeter were determined.

Table 1. Summary of micromechanical test results of Ti6Al4V alloys made using DMLS technology and milling technology from cast and forged discs

Tabela 1. Zestawienie wyników badań mikromechanicznych próbek ze stopu Ti6Al4V wykonanych w technologii DMLS oraz w technologii frezowania z dysków odlewanych i kutych

Material of tests	Location of the measurement layer	Metrology parameter	Depth of penetration [nm]	Hardness HV	Hardness HiT [MPa]	Young's modulus, E [GPa]
Samples of DMLS technology	Plane of laser beam travel	Average value	1400.6	407.6	4318.1	113.1
		Standard deviation	31.4	23.2	239.5	4.1
	Cross-section	Average value	1390,3	414,9	4395,7	111,8
		Standard deviation	15.6	12.1	128.7	2,9
Milled samples after casting and forging process	Surface layer	Average value	1489.22	344.46	3627.1	115.22
		Standard deviation	67.0	36.1	382.2	11.0

The micromechanical results indicate that the Ti6Al4V alloy from DMLS technology and milling technology from milled and forged discs has much lower values in the hardness area and the Young's modulus than the CoCrMo alloy with DMLS technology and milling technology from pressed and sintered discs **(Tabs 1** and **2)**. Ti6Al4V alloy from DMLS technology is characterized by a significantly higher hardness value – 4356.9 MPa and a slightly lower value of Young's modulus – 112.5 GPa than milling after casting and forging, for which the hardness is 3627.1 MPa and Young's modulus is 115.2 GPa. There are similar regularities in CoCrMo alloys – differences in hardness and in Young's modulus were found, depending on the production technology of the samples. The laser sintering alloy has a hardness of 6582.3 MPa and a Young's

Table 2. Summary of micromechanical test results of CoCrMo alloys made with DMLS technology and milling technology from pressed and sintered discs

Tabela 2. Zestawienie wyników badań mikromechanicznych próbek ze stopu CoCrMo wykonanych w technologii DMLS oraz w technologii frezowania z dysków prasowanych i spiekanych

Material of tests	Location of the measurement layer	Metrology parameter	Depth of penetration [nm]	Hardness HV	Hardness HiT [MPa]	Young's modulus, E [GPa]
Samples of DMLS technology	Plane of laser beam travel	Average value	1100.6	628.0	6721.1	200.3
		Standard deviation	49.8	50.1	530.0	10.0
	Cross-section	Average value	1107.3	608.2	6443.5	201.7
		Standard deviation	30.6	35.4	374.7	12.3
Samples milled after sintering with pressed powder	Surface layer	Average value	1331	470	4951	203.8
		Standard deviation	24.0	20.8	218.8	17.7

modulus of 201 GPa. Whereas, in the milling technology of the disc sintered powdered and pressed had a hardness of 4951.0 MPa and a Young's modulus of 203.8 GPa. These differences in micromechanical parameters for the CoCrMo alloy, depending on the manufacturing technology, were confirmed by tests on the Intron Wolpert TESTOR 2100 **[L. 16]**. In the micromechanical assessment of the CoCrMo alloy, there are differences in hardness, depending on the location of the plane of the laser beam movement. The test results indicate a greater hardness measured perpendicular to the planar surfaces of the structure 6721.1 MPa than the hardness determined in cross section 6443.5 MPa. The test results show a greater hardness measured perpendicular to the planes of the structure creation 6721.1 MPa than the hardness determined in cross section 6443.5 MPa. The tests and experiments prove that both alloys have sufficient micromechanical parameters, and for specific dental applications, it is advisable to modify them **[L. 1, 10, 14]**. That observation is very important when evaluating the biomaterials for manufacturing the dental structures as this innovative sintering technology can be used in prosthetics, implantoprosthetics, and facial-cranial-jaw surgery. In these varied applications, the strength parameters of biomaterials adapted for optimum biomechanical contact with pillar teeth, cortical bone, or spongy bone are required **[L. 1, 10, 11, 17–19]**.

The results of scanning test of Ti6Al4V alloys from DMLS technology and Ti6Al4V alloys from the process of the milling of cast and forged discs contained SEM images obtained from the Hitachi S3400N microscope. The sequence of Figures 4 and 6a shows selected representative SEM images of the microstructure of Ti6Al4V samples from both technologies. Ti6Al4V alloy from laser sintering technology had a two-phase, fine-grained, micro-structure of the needle-shaped and laminated character **(Fig. 4)**. The structure of the Ti64 biphasic alloy consists of a β-matrix as well as the precipitation of the α phases shaped like elongated needles. SEM images of the indents show the flash of

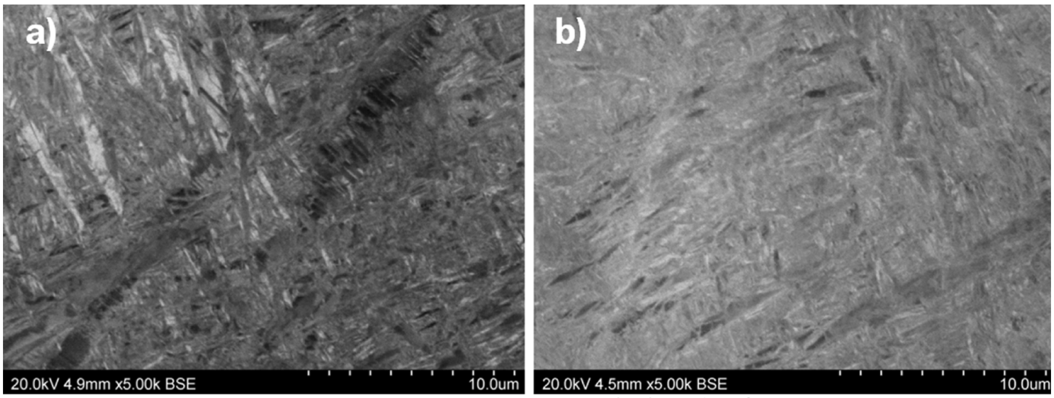

Fig. 4. SEM images of microstructure of Ti6Al4V alloy with DMLS technology: a) surface layer of the sample in the plane of laser beam movement, b) surface layer in cross section

Rys. 4. Obrazy SEM mikrostruktury stopu Ti6Al4V z technologii DMLS: a) warstwa wierzchnia próbki w płaszczyźnie przemieszczania się wiązki lasera, b) warstwa wierzchnia w przekroju poprzecznym the materials **(Fig. 7a)**. The average value of a diagonal of indent from 16 measurements is 9.89 μm. In Ti6Al4V alloy from DMLS technology, both in the plane of the laser beam moving when creating the structure and in the cross-section of this plane, the same microstructure was observed. The needle-shaped and laminated structure is not unidirectional and the needles are arranged in space in different directions. No porosity of the material was found but pores occur locally.

A granular and homogeneous microstructure is visible in the SEM images of Ti6Al4V samples milled from cast and forged discs **(Fig. 6a)**. The grains are of regular shape. The precipitates are visible at the grain boundary. In the Ti6Al4V alloy, after casting and the forging process, there is no porosity. On the SEM image of the indent, one observed the slip band (lines) in different directions on one side and the flash of the material on the other side **(Fig. 8a)**. Creation of the slip lines and flashes of materials is characteristic for the plastic material. Comparing the sizes of the indents in Ti6Al4V alloys from both technological processes, it can be noticed that, in the alloy after the milling process after casting and forging, the indent is twice as large and that indicates a lower microhardness of the alloy.

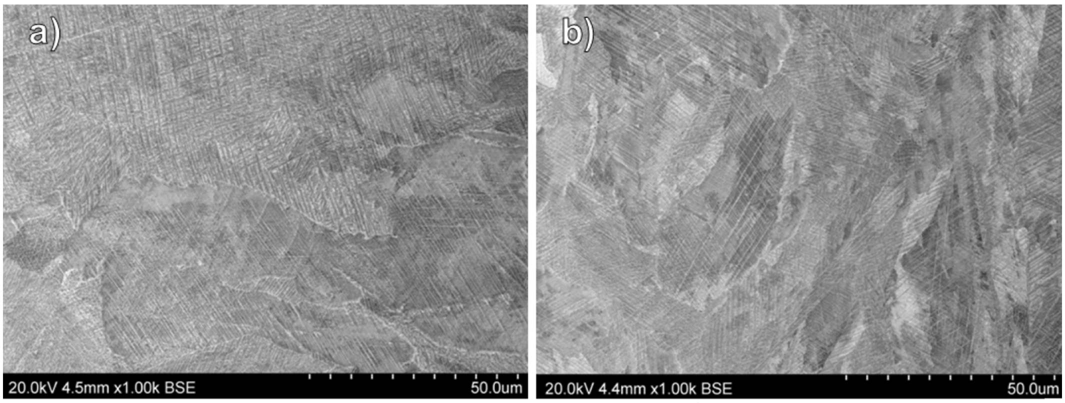

Fig. 5. SEM images of microstructure of CoCrMo alloy with DMLS technology: a) surface layer of the sample in the plane of laser beam movement, b) surface layer in cross section

Rys. 5. Obrazy SEM mikrostruktury stopu CoCrMo z technologii DMLS: a) warstwa wierzchnia próbki w płaszczyźnie przemieszczania się wiązki lasera, b) warstwa wierzchnia w przekroju poprzecznym

Based on microstructural tests of Ti6Al4V alloy from DMLS technology, it can be concluded that this material from titanium, aluminium, and vanadium contains a homogenous biphasic structure of the needle-shaped and a laminated character composed of matrix of β phase and precipitates of α phase shaped like elongated differently oriented needles **(Fig. 4)**. In the biphasic Ti6Al4V alloy, there are two important elements stabilizing the α and β phase. Aluminium is well soluble in a solid solution α, stabilizes and strengthens the α phase and causes the increase of strength. Simultaneously, the aluminium reduces the density of the alloy and increases the thermal stability of the β phase. Vanadium, belonging to the isomorphic elements, stabilizes the β phase and affects the lowering of allotropic transformation $Ti_{\alpha} \leftrightarrow Ti_{\beta}$. The microstructure of the Ti6Al4V alloy is composed of a mixture of α and β phases. A similar research profile for titanium alloys in medical applications was made by Chlebus and In. **[L. 6]**. The microstructure of the material after sintering shows the homogeneity of the chemical composition and the lack of porosity. The problem of microstructure emerged as a result of sintering of metal powders was dealt with by many authors **[L. 7–9, 17, 20, 21]**. The plate nature of the Ti6Al4V alloy microstructure has been confirmed by Ramosoeu et al. **[L. 22]** and Yadroitsev'a et al. **[L. 21]**. The two-phase microstructure of Ti6Al4V from laser sintering has also been identified by Rafi et al. **[L. 7]** and Vranken et al. **[L. 9]**. Ravi, using optical micrograms, has introduced the Ti6Al4V alloy microstructure, which consists mainly of the α phase and a small amount of β phase within the range of the prior columnar β grains oriented along the direction of the structure. The α phase has a plate-like morphology with β phase in the area of the lamellar α phase boundary.

The results of scanning tests of CoCrMo (EOS CobaltChrome SP2) alloy from DMLS technology as well as CoCrMo (Starbond CoS Soft) alloy from milling technology from sintered discs obtained from the pressed powder included SEM images from the Hitachi S3400N microscope. The sequence of **Figures 5** and **6b** shows selected representative SEM images of the microstructure of CoCrMo samples from both technologies. In the sintered structure of SP2 alloys, the scratches of the grain boundaries are visible **(Fig. 5)**. There are areas of equiaxial grains as well as grains with a strong elongated shape, which make it highly coherent. At the same time, porosity was found at the grain boundary in all analysis. Locally, there are pores within a single grain. On the SEM images of the indents, the

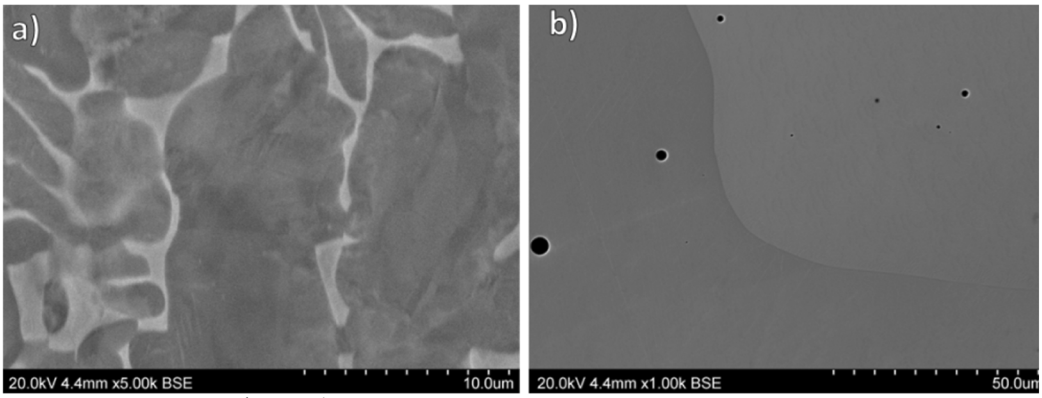

Fig. 6. SEM microstructure images: a) Ti6Al4V specimens milled from cast and forged discs, b) CoCrMo specimens milled from a powdered sintered disc

Rys. 6. Obrazy SEM mikrostruktury: a) próbek z Ti6Al4V frezowanych z dysków odlewanych i kutych, b) próbek z CoCrMo frezowanych z dysku spiekanego z prasowanego proszku

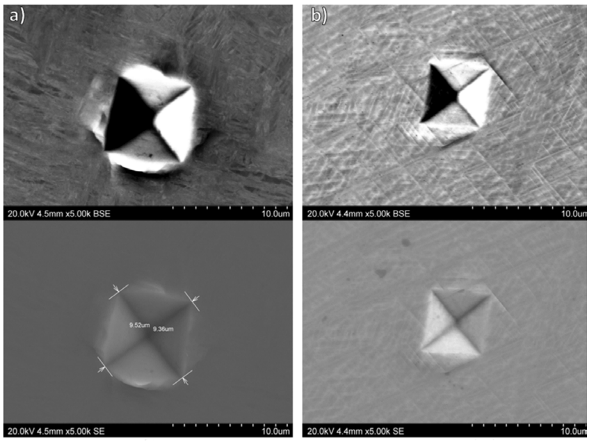

Fig. 7. SEM images (BSE and SE) of sample Vickers indentations made in samples from DMLS technology: a) Ti6Al4V, b) CoCrMo

Rys. 7. Obrazy SEM (BSE i SE) przykładowych odcisków Vickersa wykonanych w próbkach z technologii DMLS: a) z Ti6Al4V, b) z CoCrMo

occurrence of the flash of the material was not reported **(Fig. 7b)**. The dimensions of the diagonal of indent are smaller in size than in Ti6Al4V alloy. In the CoCrMo alloy from DMLS technology, both in the plane of the laser beam moving as well as in the cross-section of this plane, the same microstructure was observed.

The large grains of 200–300 μm with a clear boundary are visible in the CoCrMo alloy from milling technology of sintered discs from the pressed powder **(Fig. 6b)**. The structure is homogeneous but very small precipitates occur. The grains have a regular equiaxial shape. No flash of the material was found on the SEM images of the indents **(Fig. 8b)**. The slip lines located in 3 different directions around the indent are visible. The occurrence of the multidirectional slip lines is characteristic for plastic materials. The indents in the samples from CoCrMo alloy from the discussed technology are a bit larger than in samples from this alloy from DMLS technology. The problem of microstructure occurred when sintering metal powders was dealt with by many research centres, and the presented results were confirmed by other authors **[L. 10, 18, 23–26]**.

DMLS technology in both alloys, Ti6Al4V and CoCrMo, provides homogeneous microstructures. The indents from nanoindentation tests demonstrate it as well. It has a stable shape and lacks of flash material. The Ti6Al4V and CoCrMo microstructure after the milling process from cast and forged disc and sintered disks from pressed powder are homogeneous but show a greater graininess. The indents for both tested alloys are larger due to the lower hardness of the materials. Characteristic flashes of the material, which show much more plasticity, occur in the indents.

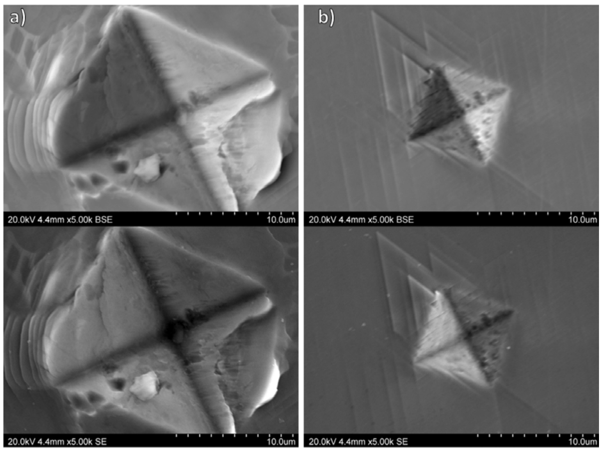

Fig. 8. SEM images (BSE and SE) of sample Vickers indentations made in samples: a) Ti6Al4V milled from cast and forged discs, b) CoCrMo milled from a sintered disc of pressed powder

Rys. 8. Obrazy SEM (BSE i SE) przykładowych odcisków Vickersa wykonanych w próbkach: a) z Ti6Al4V frezowanych z dysków odlewanych i kutych, b) z CoCrMo frezowanych z dysku spiekanego z prasowanego proszku

Greater mean friction coefficients (0.35) were found in Ti6Al4V samples from DMLS than in samples obtained by milling from cast and forged disc, for which the coefficient of friction was 0.30 **(Fig. 9)**. The mean coefficient of friction values on the level of 0.45 for samples from CoCrMo for DMLS technology were lower but close to factor for samples milled from a sintered disc from the pressed powder, which was 0.48 **(Fig. 10)**. The motion resistance in sliding friction is higher for CoCrMo specimens **(Figs. 9** and **10)** than for Ti6Al4V samples, regardless of technology of their production. The stabilization time of the coefficient of friction for Ti6Al4V samples for both technologies is very similar, which is 155 s for DMLS and 165 s for cast and forged samples. It can be stated that, for a DMLS sample, a characteristic threshold of coefficient of friction is 0.14 **(Fig. 9)**. For Ti6Al4V samples from milling cast and forged discs, an evenly increasing fluctuation of coefficient of friction to a fixed value occurred. In CoCrMo samples, the coefficient of friction stabilization time is longer and is for DMLS 230 s, and for samples milled from a sintered disc from the pressed powder it is 350 s.

Wear defects for Ti6Al4V samples from DMLS technology and milling from cast and forged discs were much greater than the wear defects for samples from CoCrMo **(Fig. 11)**. Ti6Al4V alloy from the DMLS process was characterized by slightly smaller wear (the average diameter of defects 3.18 mm) than the alloy from milling technology from cast and forged discs (the average diameter of defects 3.26 mm). The opposite phenomena occurred for CoCrMo. CoCrMo samples from DMLS had the worse wear resistance (the average diameter of defects 0.85 mm) compared to samples milled from a sintered disc from the pressed powder (0.76 mm). The tribological and wear tests of materials for dentistry applications were performed by many centres, and the results of the research were confirmed by many authors **[L. 12–14, 17, 19, 24, 27]**. Micromechanical and tribological tests show that samples of Ti6Al4V with DMLS technology include much higher hardness and slightly lower Young's modulus as well as slightly higher coefficients of friction and better wear resistance. The micromechanical and tribological tests report that CoCrMo samples from DMLS technology are characterized by much higher microhardness, a slightly lower value of Young's module, a lower coefficient of friction, and worse wear resistance.

The strength, microstructural, and tribological research were carried out on biomaterials manufactured according to standardized procedures. The reference samples included the material processes time-temperature regimes as well as equipment for CAD/CAM systems which are dedicated to dental constructions.

The application of the Ti6Al4V and CoCrMo for dental construction depends, not only on percentage and material composition, but also on the choice of technology in the area of CAD / CAM systems.

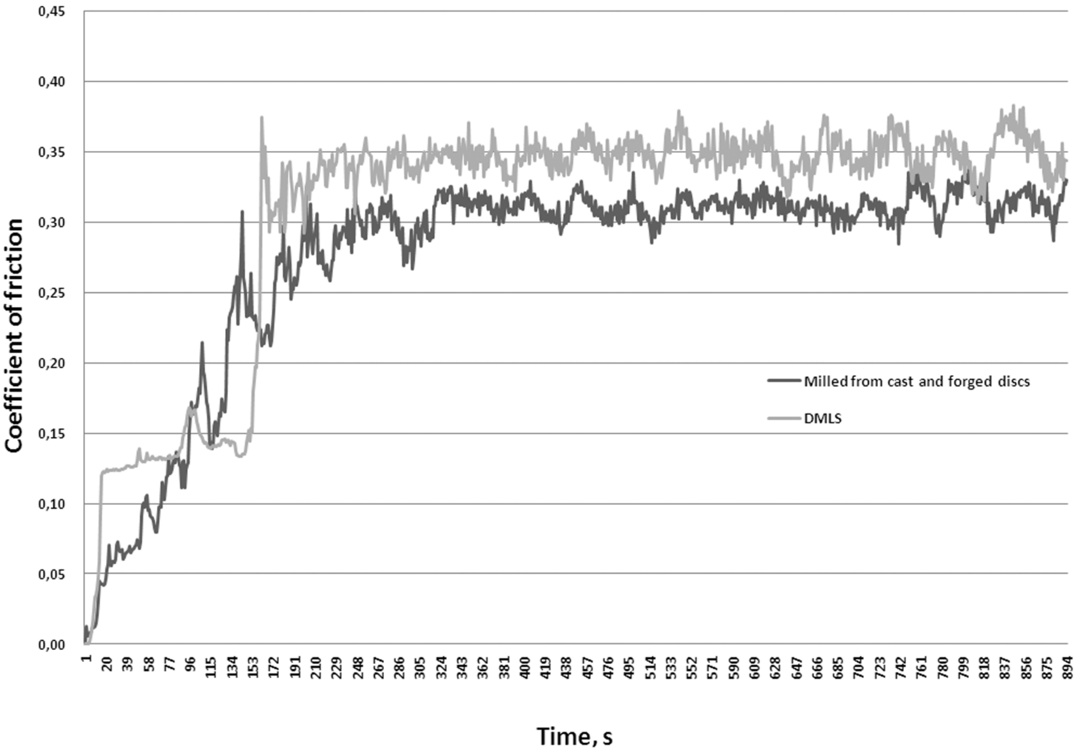

Fig. 9. Graphs of coefficients of friction for samples made of Ti6Al4V alloy in evaluated technologies

Rys. 9. Wykresy współczynnika tarcia dla próbek wykonanych ze stopu Ti6Al4V w ocenianych technologiach

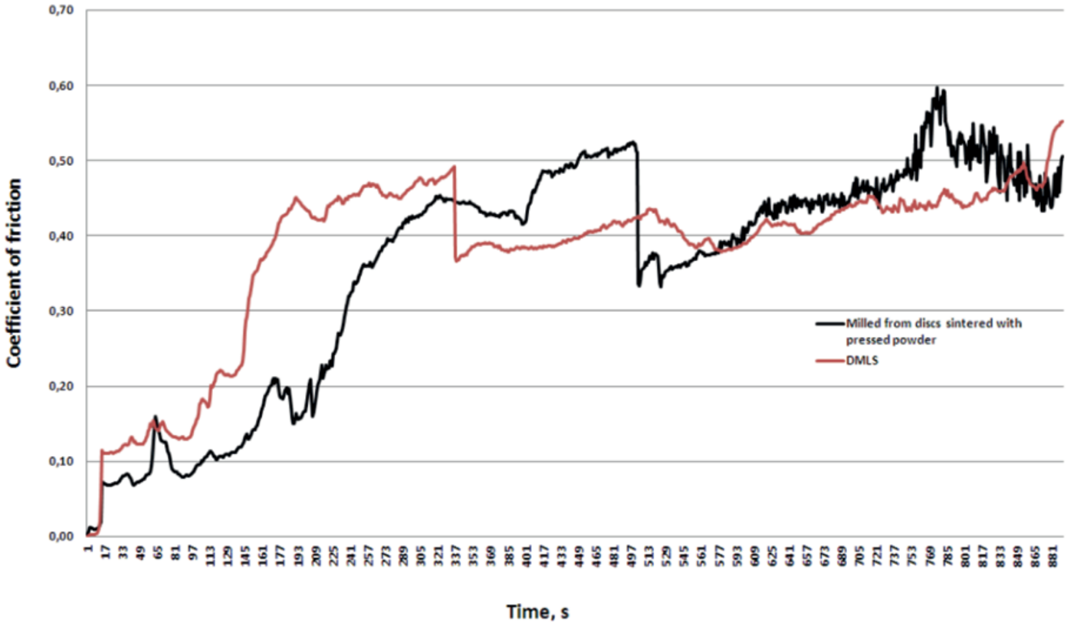

Fig. 10. **Graphs of coefficients of friction for samples made of CoCrMo alloy in evaluated technologies**
Rys. 10. Wykresy współczynnika tarcia dla próbek wykonanych ze stopu CoCrMo w ocenianych technologiach

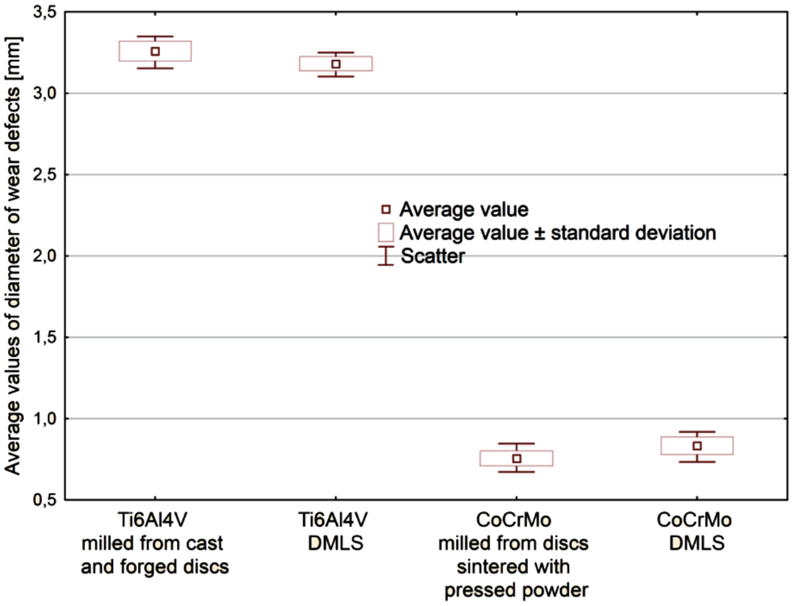

Fig. 11. **The comparison of wear resistance test results**
Rys. 11. Zestawienie wyników badań odporności na zużycie

CONCLUSIONS

The conducted research show that, in the DMLS as compared to milling technology preceded by casting and forging processes and the pressing of the powder and sintering, micromechanical, microstructural and tribological properties change for the same composition of elements.

In the range of micromechanical parameters of Ti6Al4V and CoCrMo samples from DMLS, the hardness increase and slight decrease of the Young's modulus were stated.

The microstructural studies of Ti6Al4V alloy and CoCrMo alloy from DMLS technology show their optimum granularity, very few porosities, and homogeneity.

In samples from Ti6Al4V from DMLS, wear resistance slightly improved and the coefficient of friction increased. CoCrMo samples from DMLS show a slight decrease in wear resistance and an insignificant decrease in the coefficient of friction.

On the basis of the research, it can be stated that incremental laser sintering from selective powders, in terms of material structure and mechanical parameters, is a new technology that is preferred for the construction of supporting structures for dentistry. It can be an alternative to traditional methods based on traditional moulding and CAD/CAM based milling. This technology does not generate material loss characteristic to milling and is environmentally friendly.

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