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EFFECT OF THE SHOT PEENING ON SURFACE PROPERTIES OF Ti-6Al-4V ALLOY PRODUCED BY MEANS OF DMLS TECHNOLOGY

WPŁYW NAGNIATANIA STRUMIENIOWEGO NA WŁAŚCIWOŚCI EKSPLOATACYJNE STOPU Ti-6Al-4V UZYSKANEGO TECHNOLOGIĄ PRZYROSTOWĄ DMLS*

The state of the surface layer and biocompatibility are the key parameters contributing to successful implantation of prostheses such as bone implants which are now increasingly often produced by means of DMLS technologies. The analysis of these factors and proper selection of material are required in order to determine the most favourable technological parameters contributing to long term functioning in course of their presence in human body. Therefore, the purpose of the present paper is to investigate the effect of shot peening on the state of the surface layer and corrosion resistance of specimens made of Ti-6Al-4V titanium alloy produced in Direct Metal Laser Sintering (DMLS) process. The specimens have been produced by means of EOSINT M280 system dedicated for laser sintering of metal powders and their surfaces have been subjected to the shot peening process under three different working pressures (0.2, 0.3 and 0.4 MPa) and by means of three different media i.e. CrNi steel shot, crushed nut shells and ceramic balls based on ZrO_2. It has been found that the process conditions i.e. working pressure in course of shot peening and proper selection of applied shot will make it possible to achieve the properties in modified material sufficient to ensure that assumed functions associated with the improvement of surface layer condition are invariable during required period in specified implant operation conditions. In such case, these factors have been determined in course of microhardness tests, evaluation of surface development degree as well as potentiodynamic tests. The increase of working pressure caused deteriorated corrosion resistance. Simultaneously, it has been found the corrosion resistance was most satisfactory for the surfaces modified by means of: ceramic balls based on ZrO_2 > crushed nut shells > CrNi steel shot correspondingly.

Keywords: *additive manufacturing, shot peening, exploitation, titanium alloys, corrosion resistance.*

Stan warstwy wierzchniej i biozgodność to podstawowe czynniki mające wpływ na efektywną implantację. W przypadku wyrobów medycznych takich jak: implanty kostne, które obecnie są coraz częściej wytwarzane z wykorzystaniem technologii przyrostowych, analiza tych czynników oraz właściwy dobór materiału jest niezbędny do określenia najbardziej korzystnych parametrów technologicznych przyczyniających się do długotrwałego działania podczas ich eksploatacji w organizmie. Dlatego też celem niniejszego artykułu jest zbadanie wpływu obróbki nagniataniem strumieniowym (ang. shot peening), na stan warstwy wierzchniej i odporność korozyjną próbek ze stopu tytanu Ti-6Al-4V wytworzonych technologią przyrostową DMLS (Direct Metal Laser Sintering). Przy zastosowaniu systemu laserowego spiekania proszków metali EOSINT M280 wykonano próbki, których powierzchnie następnie poddano obróbce nagniatania strumieniowego przy trzech rożnych ciśnieniach roboczych (0.2, 0.3 i 0.4 MPa) z wykorzystaniem trzech różnych mediów tj.: śrutem ze stali CrNi, rozdrobnionymi łupinami orzechów oraz kulkami ceramicznymi na bazie ZrO_2. Stwierdzono dla wszystkich badanych powierzchni, że warunki procesu tj. ciśnienia roboczego obróbki nagniataniem strumieniowym oraz odpowiedni dobór śrutu pozwolą na uzyskanie takich właściwości w modyfikowanym materiale, że założone funkcje poprawy stanu warstwy wierzchniej będą niezmienne przez wymagany czas w określonych warunkach eksploatacji implantu. W tym przypadku wskaźniki te określono podczas badań mikrotwardości, oceny stopnia rozwinięcia powierzchni oraz testów potencjodynamicznych. Wzrost ciśnienia roboczego powodował pogorszenie odporności na korozję. Przy czym najbardziej korzystnie pod względem odporności korozyjnej zachowywały się powierzchnie modyfikowane kolejno: ceramiką na bazie ZrO_2> łupinami orzechów> śrutem stalowym CrNi.

Słowa kluczowe: *wytwarzanie przyrostowe, nagniatanie strumieniowe, eksploatacja, stopy tytanu, odporność korozyjna.*

1. Introduction

Titanium and titanium alloys belong to the group of most important metallic materials which are currently used for implants in orthopedics and in dental surgery [7, 10].

Additive manufacturing currently belong to the most promising methods of Ti-6Al-4V alloy processing for biomedical applications, particularly in view of metallic materials manufacturing with consideration of the requirements to be met for individual patients [8]. According to data available in literature [1, 5], the surface modifi-

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(*) Tekst artykułu w polskiej wersji językowej dostępny w elektronicznym wydaniu kwartalnika na stronie ***www.ein.org.pl*** cation has a definite influence on exploitation conditions of medical implants. Insufficient quality of surface finishing in combination with wearing and corrosion processes contribute, among other things, to reduced service life of implants and increased number of necessary revision surgeries [10, 17].

In recent years, we have seen intensive development of techniques associated with precise processing of the products obtained by means of additive manufacturing. The results presented in literature [14, 15, 21] enable an effective choice of processing parameters for such products. Moreover, the manufacturers of materials obtained as a result of DMLS technologies suggest various types of finishing processes e.g. cleaning, shot blasting or abrasive techniques. Direct Metal Laser Sintering technology itself causes the generation of residual stresses in the product [12]. The surface peening by means of shot belongs to the methods used to improve the operational properties of machine components [25]. In the opinion of Benedetti et al. [4], the surface hardening by means of shot blasting introduces favourable compressive stresses and reduces the porosity of surface layers which subsequently translates into the increase of fatigue strength. The manufacturers of laser sintering systems for technical products recommend the shot peening as the process improving operational properties of the product surface layer. However, there are no detailed technological guidelines for such kind of treatment i.e. size and shape of shot grains or shot type, working pressure or kind of material used for shot production. Generally, the increase of shot grain size at constant working pressure usually caused the reduction of roughness [23]. However, it is not an explicit statement because in the tests [6] the increase of grain size from 125÷250 μm to 450 μm caused increased roughness but its reduction was achieved at double size grain 850 μm. reduced size of shot grain causes the reduction of indentation size after shot peening process. Therefore, the number of indentations per surface unit will be increased.

Most often for the products obtained as a result of additive manufacturing applied in engineering (mainly in aircraft industry) are used, among others: CrNi steel shot, nutshells or ceramic beads. After the shot peening process, shot particles can penetrate into superficial layer (as permanent deposit) and change corrosion resistance of the products being modified [1]. Therefore, all experimental studies associated with assessment of the technical condition of products contribute to the improvement of their durability [19]. However, there are no explicit guidelines in literature concerning the surface layer treatment technology with consideration of high technological regimes for medical products depending on intended use of implant and any information about reliability of their functioning in human body is also unavailable.

Titanium and titanium alloys are characterized by high corrosion resistance due to passive layers created on their surfaces and mainly consisting of amorphous TiO_2 [10, 11, 13]. The stability of passive layers in various biological environments in combination with favourable physicochemical properties of oxide passive layers translates into biocompatibility of titanium implants [1, 4]. Generally, the majority of material damages, including stress cracks, abrasive or corrosive wear is generated on the product surface. Therefore, recently many studies were concentrated on the solution of this problem. It has been found that the proper modification of surface material layer can lead to the improvement of the material behaviour in an environment affected by variable load and the influence of body fluids and consequently to the improved functioning reliability.

In view of all the above, the subject matter of our research encompassed the analysis of proper shaping of surface layer of titanium products obtained by means of direct laser sintering of metal powders in the form of DMLS technology.

2. Material and tests methodology

2.1. Specimens preparation

The specimens have been made of gas atomized powder of Ti-6Al-4V alloy with chemical composition meeting the requirements of ASTM F136 in the scope of maximum content of impurities. The specimens shaped in the form of cubes with dimensions of 10 x 10 x 10 mm have been made by means of DMSL technique using EOSINT M280 system (EOS GmbH, Germany) dedicated for laser sintering of metal powders. The most important printing parameters are, among other things, distance between paths of 0,1 mm, laser beam speed of 1250 mm/s, the thickness of melted powder layers of 30 μm and applied power of laser beam of 170 W.

Outer surfaces of specimen face in horizontal plane X-Y has been subjected to shot peening process on Peenmatic micro 750S device (IEPCO, Switzerland) by means of three different media i.e. CrNi steel shot, nutshell granules and ceramic beads based on ZrO_2. Three different working pressures have been applied: 0.2, 0.3 and 0.4MPa. The time of surface treatment was equal to 60 s and the distance between the nozzle and the face of surface being processed ~25 mm. The shot peening process was carried out perpendicularly to the surface.

Principal parameters of the materials used in the shot peening process are included in table 1. The characteristics of the materials used in the shot peening process have been determined by means of electron scanning microscope Quanta 650 (FEI, Holland) and microphotographs are presented on Fig. 1.

The products obtained directly after sintering (without surface modification) and specimens subjected to grinding and polishing have been used as reference specimens. Additionally, the results have been compared with specimens subjected to mechanical polishing, because standard specimens with high surface quality (usually Sa<0.05μm) are very often used in literature for the materials produced as biomaterials (particularly in dental prosthetics) for their corrosion resistance evaluation. Therefore, in the first place, the specimens have been subjected to grinding on grinding and polishing machine Saphir 530 (ATM, Germany) using water papers with grain size of 200, 600, 800 and 1200 correspondingly. Then the specimens were subjected to me-

Table 1. Parameters of shot for shot peening

Shot	Typical chemical composition (%)		Average grain size (μm)	Grain shape	Hardeness
Stainless steel shot – CrNi	Cr Ni Si Mn C Fe	16-20 7-9 1.8-2.2 0.7-1.2 0.05-0.2 bal.	400-900	spherical	235HV
Nutshell granules	non-ferrous, organic blasting media		450-800	angular	approx. 2.5-3.5 Mohs
Ceramic beads	ZrO_2 SiO_2 Al_2O_3 CaO TiO_2 Fe_2O_3	61.98 27.77 4.57 3.47 0.34 0.14	125-250	spherical	approx. 7-7.5 Mohs

chanical polishing using slurry of diamond 3 μm and slurry of oxides 0.04 μm as well as to washing with acetone and drying in warm air stream after completed polishing. The grinding and polishing process was carried out at the speed of 240 and 120 rpm correspondingly and the head pressure was equal to 27N.

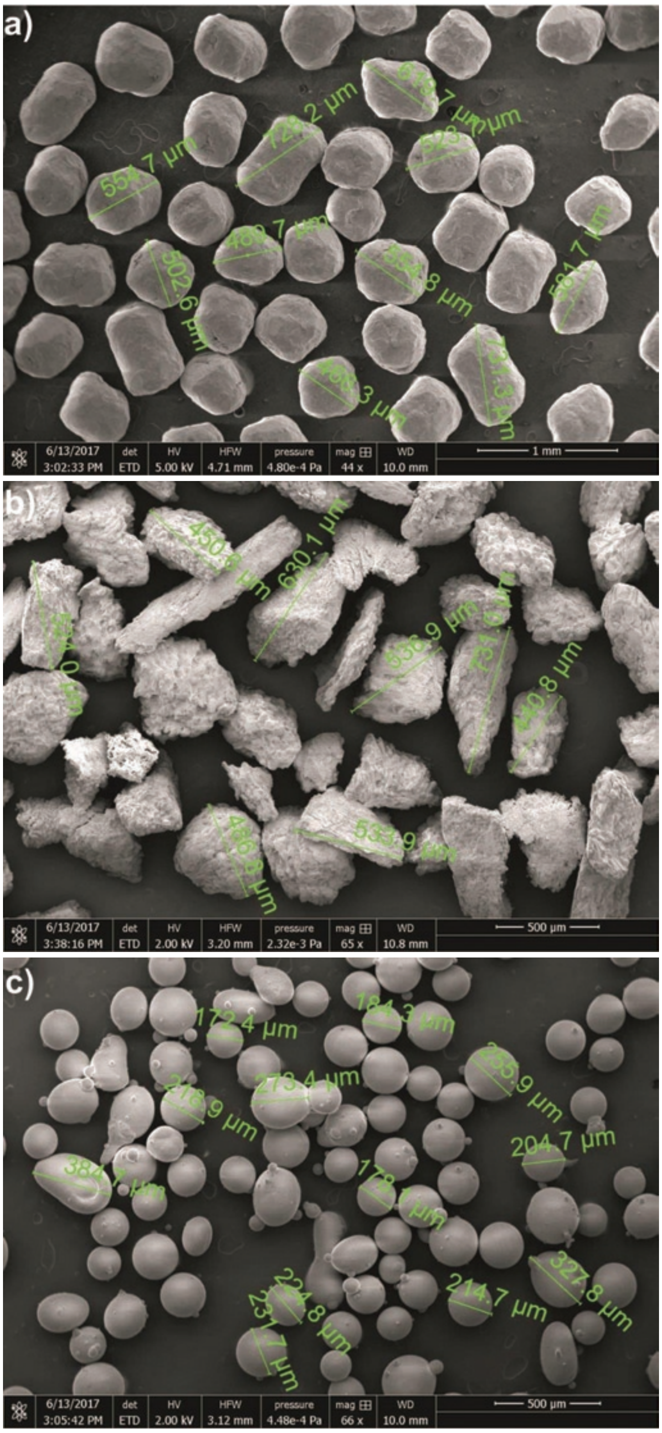

Fig. 1. SEM microphotography of shot: a) CrNi steel, b) nutshells and c) ceramic beads

2.2. Test methodology

The specimens to be tested have been subjected to profilometric measurements on Contour GT optical profilometer (Bruker, Germany). The measurements were carried out under magnification 5.5x. The profilometric analysis encompassed the surface area of 5 mm x 5 mm using VSI method (Vertical Scanning Interferometry) and obtained signals have been converted by means of Bruker Vision 64 software.

The degree of material hardening has been measured by means of Vicker's FM-700 microhardness tester with ARS 900 automatic system (Future-Tech Corp., Japan) on perpendicular metallographic samples 30 μm from the surface contour edge. The measurements were carried out under HV0.05 load (50g). Ten (10) hardness measurements have been completed for each group of specimens.

The susceptibility to corrosion has been determined for specimens being tested by means of accelerated electrochemical tests in 0.9% NaCl solution at temperature of 37 °C using Atlas 0531 set dedicated for corrosion tests. The tests were carried out in an electrochemical vessel with three electrodes i.e. with platinum electrode applied as control electrode and saturated calomel electrode (SCE) applied as reference electrode. The surface area of electrode being tested was equal to 1 cm^2. Polarization curves have been recorded with an automatic potential shift of 1 mV/s in the range between –800 mV and +2000 mV. The values of density of corrosive currents i_{corr} and potentials E_{corr} have been determined from Tafel curves thanks to potentiodynamic curves analysis in AtlasLab program.

3. Results and discussion

3.1. Geometrical structure of surface

The process of products manufacturing in DMLS technology takes place along the laser beam scanning direction on the surface of thin layer of powder deposited on the substrate (base plate). Therefore, as a result surface tension of melted material, a laser melted track is created (Fig. 2a) which represents the laser operation direction. The laser beam penetration into substrate or to the previously sintered layer provides an additional stabilizing effect for continuous creation of paths [23] but excessive penetration of keyhole is impermissible in DMLS technology, because such phenomenon can generate the pores in the final 3D object due to collapse of welding puddle or occurrence of structural discontinuities in the form of gas bubbles in material [18, 20]. In the opinion of Yadroitsev et al. [24] a deeper penetration of laser beam (the *depth* of weld penetration) penetrating significantly deeper than the thickness of sintered layer is also undesirable for energy reasons.

Additionally, on the outer surface in X-Y plane (Fig. 2b) it is possible to observe not wholly melted titanium powder grains. In case of untreated surfaces such grains can act as micro-notches and, under the influence of external load, constitute the areas of potential development of micro-cracks situated in surface layers which consequently reduce strength parameters of the whole product.

As described in the literature [1, 4, 6], Sa – arithmetic average of surface roughness is used for the evaluation of machined surfaces as the most representative parameter for surfaces after shot peening. On the basis of analysis of the results obtained from profilometric measurements Fig. 3 and table 2, it is possible an increase of surface development with the increase of working pressure in the shot peening process for all modified surfaces. The studies [3, 6] confirm that the increase of working pressure at constant shot size leads to roughness increase.

Generally, the shot peening process by means of CrNi steel shot as well as by means of ceramic beads causes the reduction of roughness in relation to unmodified surface (obtained directly after DMLS sintering). In case of surface modification by means of nutshell granules, observed roughness increase is greater than in case of an unmodified surface. Such behaviour is probably caused by irregular sharp-edged shape of used grains while CrNi steel shot and ceramic beads were almost spherical. The traces of laser sintering (directions of laser beam movement) can be observed on Fig. 3 only for surface modified by means of nutshell granules.

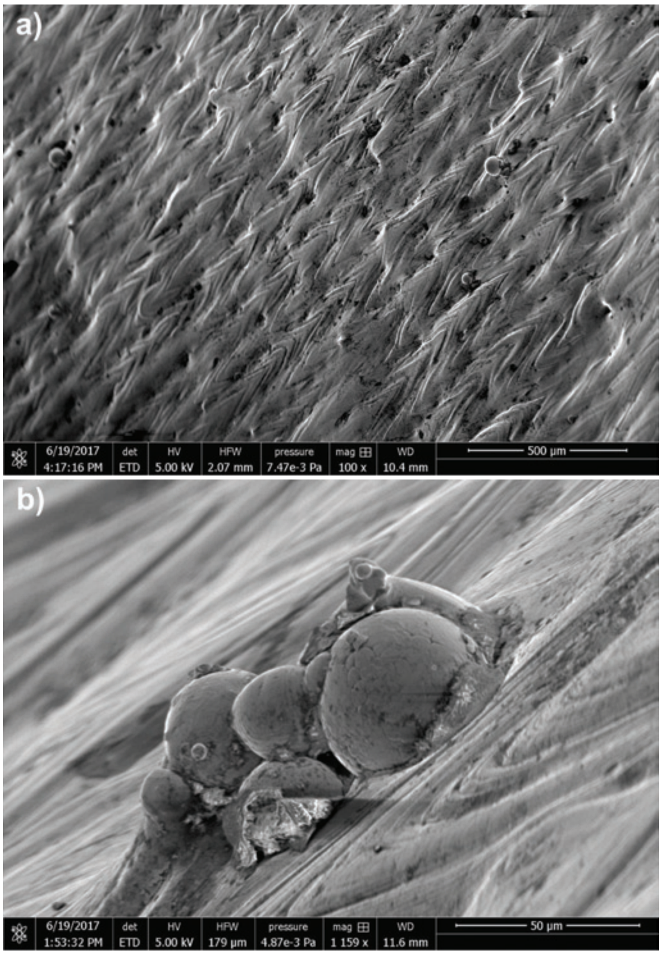

Fig. 2. SEM microphotographs illustrating the morphology of specimens surface after sintering by means of DMSL technology: a) visible laser beam scanning directions, b) partially melted titanium powder grains.

It is worth noting that at lower average size of ceramic beads (almost three times) in comparison to CrNi steel shot at working pressure of 0.2 MPa it is possible to achieve higher roughness parameters Sa (Table 2). Small shot grain size causes reduced indentation size after shot peening process but it is translated into increased number of indentations per surface unit.

However, the differences in the value of Sa parameter between the surfaces tread by means of CrNi steel shot and ceramic beads correspondingly and the pressures of 0.3 and 0.4 MPa are not significant. The amount of shot hitting the surface being treated is increased at higher pressure and the reinforcement of shot material (reduction of grain size and hardness increase) takes place in case of CrNi steel shot. In the opinion of Kuhmichel, double increase of grain hardness is possible from 235HV to 460HV. The hardness of ceramic beads is much higher than the hardness of titanium and its geometry in course of shot peeling proces is not changed. Therefore, the average increase of roughness parameter Sa by about 0.3÷0.4 μm is observed at constant increase of working pressure by 0.1 MPa.

Table 2. Summary of average arithmetical deviation of roughness Sa(μm) for surfaces being tested

Peening pressure (MPa)	Steel CrNi	Nuts	Ceramics	Unmodified surface after DMLS	Mechanicallypolished
0.2	5.015	7.641	6.106	7.483	0.039
0.3	6.381	7.829	6.595		
0.4	6.904	7.877	6.953		

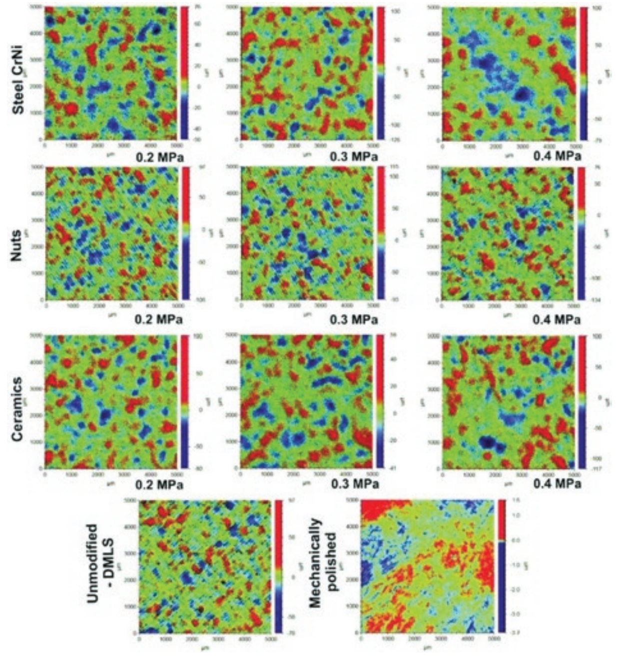

Fig. 3. Maps illustrating roughness for various surface modifications (scan size 25 mm²)

Titanium belongs to materials characterized by high ductility and plasticity with the trend towards spontaneous oxidation. It is therefore possible that shot grain will penetrate (insert) into modified surface layer causing additional change of treated surface topography. Particularly visible are the residuals of nutshells on treated surface in the form of dark areas. Furthermore, in case of shot blasting process by means of ceramic beads and CrNi steel at low pressured (Fig. 4) it is possible to ensure more uniform surface in comparison with the surface obtained directly after DMSL sintering and to introduce small impact indentations which is also confirmed by the roughness maps presented on Fig. 3.

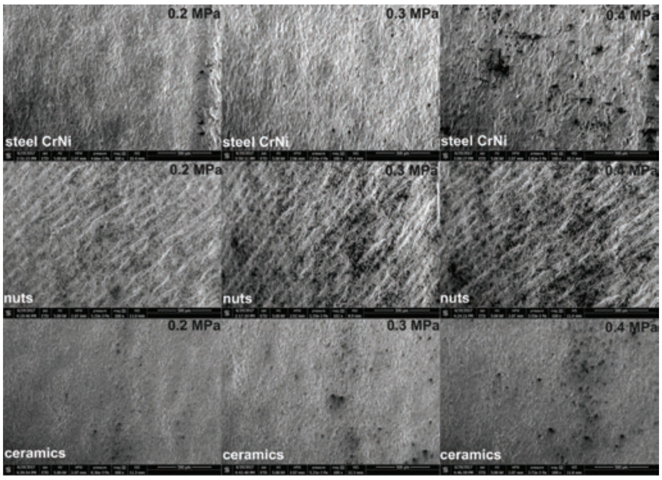

Fig. 4. SEM microphotographs illustrating the morphology of specimens surface. Magnification x100

3.2. Microhardness

Microhardness testing (Fig. 5) demonstrated the increase of average hardness values for all the treated surfaces in comparison with specimens obtained directly after DMSL sintering. The strengthening of surface layer has been achieved with the increasing pressure for all the variants of surface modification. In the opinion of Ahmed et al. [1], titanium reinforcement is possible to the depth of 0.1÷0.8 mm depending on shot peening process parameters. However, in the opinion of Benedetti et al. [4], the hardness of superficial layer in modified titanium alloy can reach the depth of 0.15÷0.3 mm while the largest changes have been observed by the authors [1] for the depth of about 70 µm but, despite noticeable differences in microhardness, obtained results were not statistically significant.

The average hardness of specimens produced in DMLS process was equal to 334HV i.e. was slightly higher than the value declared by the manufacturer GmbH – 320HV. The smallest increase of hardness in relation to reference samples (DMLS) has been achieved for surfaces modified by means of nutshell granules. However, extremely comparable results have been obtained in course of treatment by means of steel shot and ceramic beads. The values of microhardness for surfaces treated by means of ceramic beads have been achieved at less than half the size of shot grains.

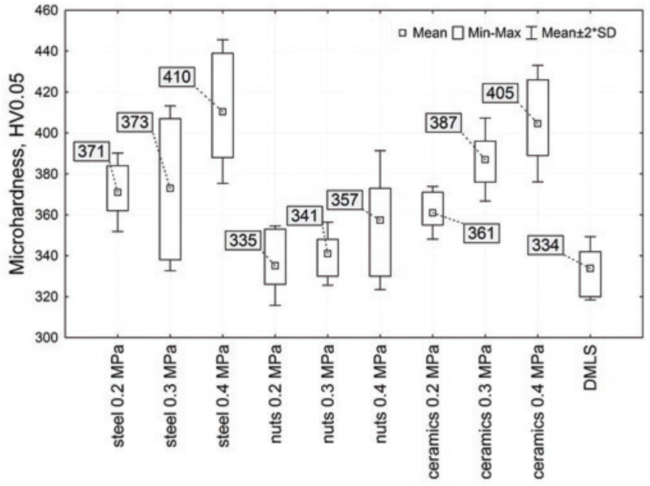

Fig. 5. Results of microhardness measurements for various surfaces.

In order to verify whether achieved changes are statistically significant, the analysis has been carried out by means of STATISTICA program using parametric tests for independent tests. On the grounds of statistical analysis by means of Shapiro-Wilk test for microhardness measurement, it has been demonstrated that obtained results do not have a normal distribution $p<0.05$ (assuming $\alpha=0.05$). Therefore, $p<\alpha$ and there are the grounds for rejection of the hypothesis of normal distribution for the feature being tested. Owing to the fact that the values obtained in course of microhardness testing did not have normal distributions, the results of nonparametric tests for Kruskal-Wallis independent tests (for $\alpha=0.05$) have been applied for statistical analysis. On the grounds of statistical analysis it has been demonstrated that the differences in reinforcement of surface layer are statistically significant ($p<0.05$) only between selected groups of surfaces being tested i.e. steel (0.4 MPa) and nutshells (at the pressure of 0.2 and 0.3 MPa), nutshells (0.2 MPa) and ceramic beads (0.4 MPa). However, in case of comparison with reference group (DMLS), statistically significant differences have been observed for surfaces modified by means of steel shot and ceramic beads at the pressure of 0.4 MPa only.

3.3. Corrosion behaviour

The evaluation of the surface layer from operational aspects can be precisely described by means of electrochemical corrosion tests. The results of potentiodynamic polarization testing for surfaces with different modification degree have been presented in table 3 and on Fig. 6. From analysis of Tafel polarization curves it appears that the increase of pressure in shot peening process causes deteriorated corrosion resistance of surfaces being tested which manifests itself in the form of reduction of corrosion potential E_{corr} while the lowest impact has been observed for ceramic beads. The decrease of corrosion resistance is associated with the increase of surface development (roughness profile) after the shot peening process. The decrease of corrosion resistance is associated with the increase of surface development (roughness profile) after the shot peening process. Therefore, in case of surfaces treated by means of nutshell granules with the highest Sa roughness parameters, the worst E_{corr} values have been obtained. Nevertheless, the surfaces modified by means of ceramic beads and nutshell granules are characterized by the trend towards creation of a permanent passive layer firmly adhering to the surface (passivation ability). However, in case of surfaces modified by means of CrNi steel shot, there are distortions in passive area demonstrating the trend of material towards the building of a passive layer without firm attachment to the substrate. Such situation is probably caused by CrNi shot grains deposited (after shot peening process) in surface layer and hindering passivation process. Furthermore, at the potential value of about 1.5V, transpassivation ability is observed for the surfaces modified by means of ceramic beads and nutshell granules only. Therefore, from among for the surfaces modified in shot peening process preferably in 0.9% NaCl environment (in medical application context) the best are for the surfaces modified by means of ceramic beads > nutshell granules > CrNi shots successively. On the grounds of comparison of electrochemical parameters to reference group (specimen marked as DMLS), the most favourable values (the lowest i_{corr} and the highest E_{corr}) have been obtained for the surfaces modified by means of ceramic beads. Even in case of penetration of ceramic beads into superficial layer, they will act as an insulating material neutral in terms of biocompatibility.

However, from among the materials subjected to tests, the best electrochemical parameters have been observed for mechanically polished surfaces. They had most favourable electrochemical parameters (table 3), and Tafel curve (Fig. 6d) was characterized by the wide passive area. However, the results obtained in this testing for unmodified surfaces after 3D printout in DMLS technology were slightly worse. Comparing the surfaces of reference

Table 3. Results of electrochemical corrosion tests for various surfaces

Conditions		Current density, i_{corr} (nA/cm^{-2})	Potential, E_{corr} (mV)	Polarization resistance R_p (kΩcm^2)
steel CrNi	0.2 MPa	420.9	–173.5	210.5
	0.3 MPa	560.7	–207.4	138.8
	0.4 MPa	682	–337.1	81.2
nuts	0.2 MPa	123.8	–106.6	346.5
	0.3 MPa	275.3	–228.5	367.4
	0.4 MPa	1469.8	–279.2	349.5
ceramics	0.2 MPa	26.3	–123.8	170.8
	0.3 MPa	44.8	–151.4	206.2
	0.4 MPa	63.1	–174.3	432.8
DMLS		64.3	–318.6	2291
mechanically polished		67.3	–141.1	328.5

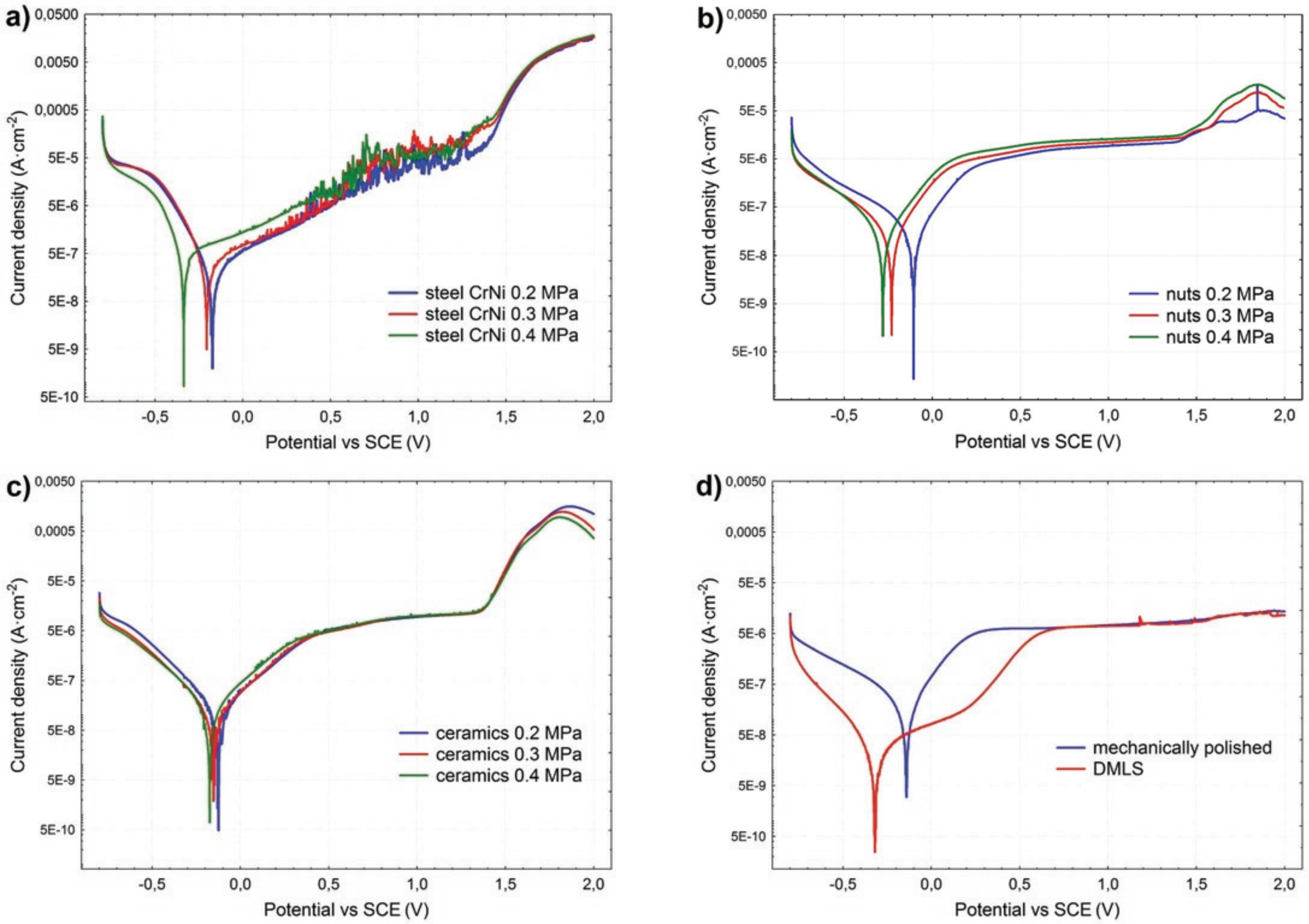

Fig. 6. Potentiodynamic corrosion tests for surfaces modified by means of various methods: a) CrNi steel shot, b) ceramic beads c) nutshell granules, d) reference surface and surface after mechanical polishing

specimens with polished specimens, we can found that the polished surface quality is better in terms of corrosion resistance because it is characterized by lower values of Sa parameter and the lack of structural discontinuities in surface layer created in course of DMLS process (see Fig. 2).

In accordance with literature analysis, Ahmed et al. [1] obtained favourable results in shot peening process by means of ceramic beads for increasing shot diameter. However, this conclusion is not obvious because in accordance with data [16, 22] it has been found that the decrease of grain size can lead to increased corrosion rate but, on the other hand, some publications [2, 9] demonstrated improvement of corrosion resistance at reduced grain size. However, in case of steel shot there are literature data [10] indicating favourable results in the scope of corrosion resistance in environment simulating body fluids in comparison with unmodified surfaces. But these tests have been carried out for titanium produced in conventional process and the diameter of balls made of 100Cr6 bearing steel applied in this process was equal to 1÷8 mm i.e. significantly more than in case of presented tests. The phenomenon of shot penetration into the surface layer of material being treated does not occur in case of such large sizes of steel shot. Therefore there no disturbances are observed in the passive area of Tafel curve in course of the creation of permanent oxide layer.

4. Summary and conclusions

The tests of the products made of Ti-6Al-4V powder in DMLS process and subjected to shot peening process thereafter showed that in case of properly selected technology it is possible to improve the exploitation properties of implants. Owing to the fact that the shot peening process was carried out at three different pressure values: 0.2, 0.3 and 0.4 MPa, it was possible to analyse the effects of tests in the context of reliability evaluation for the products characterized by high quality of treated surfaces and dedicated for medical applications. Therefore, except of surface topography analysis, SEM observation, microhardness measurements, properly modified specimens have been subjected to corrosion resistance evaluation in 0.9% NaCl environment. The following conclusions have been drawn from the tests:

- Pressure increase in course of shot peening process for all the groups of materials being tested leads to the reduction of corrosion potential associated with the increase of surface development and the presence of CrNi steel shot in surface layer causes the disturbance of the material passivation process.
- The shot peening process by means of CrNi steel shot as well as by means of ceramic beads leads to the reduction of roughness in comparison with unmodified surface obtained from DMLS technology. However, in case of treatment by means of nutshell granules observed roughness increase was slightly greater than in case of unmodified surface.
- Pressure increase in course of shot peening process causes the increase of surface roughness.
- The shot peening process by means of CrNi steel shot as well as by means of ceramic beads leads to the increase of microhardness in surface layer of the material being treated while in comparison with unmodified surface the most statistically significant differences have been observed for surfaces modi-

fied by means of steel shot and ceramic beads at the pressure of 0.4 MPa only.

- The highest corrosion resistance in 0.9% NaCl environment (in the context of medical applications) has been found for the surfaces modified by means of ceramic beads > nutshell granules > CrNi shot successively. The most favourable electrochemical parameters (much better than in case of unmodified specimens) have been obtained for surfaces treated by means of ceramic beads and the parameters for nutshell granules have been found acceptable. The next phase of tests should be carried out in the form of in vivo studies in order to verify the suitability of nutshell granules for applications of personalized implants production meeting the individual needs of the patients.

Currently there are no information in literature concerning the suitability of nutshell granules for the use in shot peening process for the products obtained from DMLS processes and dedicated for medical applications. In spite of satisfactory results in the scope of corrosion resistance obtained for surfaces treated by means of such medium, it would be worth to check the behaviour of surfaces treated by means of nutshell granules in vivo environment in further tests.

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