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

The purpose of the present paper was to investigate the effect of shot peening on the condition of the surface layer and abrasion resistance of specimens made of Ti-6Al-4V titanium alloy produced by 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. The specimens have been subjected to profilometric analysis, to SEM examinations, microhardness tests and to tribological tests on ball-on-disc stand in Ringer fluid environment. The general results of all tests indicate to favourable effect of shot peening process on the hardness and tribological performance of titanium alloy.

Keywords: additive manufacturing, shot peening, titanium alloys, wear, tribology

1. Introduction

Thanks to favourable properties e.g. high biotolerance, excellent mechanical properties and high corrosion resistance in the environment consisting of tissues and body fluids, titanium and its alloys are commonly applied in medicine and particularly in the field of implantology and dental prosthetics [1-3]. However, defects of metal implants are observed in case of vitro conditions which are mainly caused by surface layer [4,5]. Poor wear resistance of titanium alloys or insufficient finishing quality of implant surface in course of long term service can consequently lead to its damage which is a serious clinical problem associated with the necessity of revision surgeries. Even relatively small wear combined with the impact of body fluids environment can accelerate the process of metal ions release and leads to the reduction of implant service life due to unfavourable reactions with tissues.

Titanium implants with complex shapes can be produced by conventional processes in the form of casting, material removal, plastic forming or powder metallurgy [6-8]. Nevertheless, intensive growth of additive technologies is observed lately [6,9]. At the moment, additive technologies are perfectly suited to the cases when it is necessary to produce so called personalized implants. In such case, the adaptation of specified implant to anatomic features of the patient can have decisive influence on the success of implantation surgery.

The manufacturers of metal powder sintering systems for products manufactured in 3D printing process recommend various types of finishing processes e.g. cleaning, shot blasting or abrasive techniques. However, the use of laser sintering technology leads to the creation of residual stresses in products [9,10]. Shot peening process is particularly recommended for this type of products. As a result of shot peening process, favourable compressive stresses are introduced into surface layer and fatigue strength of metal elements is increased [10]. According to data available in literature [4,11], the plastic strain leads to nanocrystallization in surface layer which has a favourable influence on corrosion resistance and wear except of strength increase. It has been suggested that dislocation density and shot size can affect the properties of surface layer of the products being modified. Whereas, in accordance with our own research and information available in literature [12,13] as a result of shot peening process, the shot grains can penetrate to surface layer (permanently depositing) and modify tribological performance and corrosion resistance in the products being modified in this way. The products manufactured by means of additive technologies and dedicated for technical applications e.g. in aircraft industry are very often modified by means of steel shot, crushed nut shells or ceramic balls. In case of products fabricated for medical applications characterized by high level of technological regime, there are no defined guidelines applicable to forming of surface layer in such products. Therefore, the purpose of the present

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paper is to examine the effect of shot peening process on the condition of surface layer and abrasion resistance of specimens made of Ti-6Al-4V titanium alloy produced by Direct Metal Laser Sintering (DMLS) process.

2. Materials and methods

The specimens have been made of gas atomized powder of Ti-6Al-4V alloy with almost spherical shape and chemical composition meeting the requirements of ASTM F1472 (Fig. 1a) in the scope of maximum content of impurities. The specimens shaped in the form of discs with diameter of 30 mm and thickness of 6mm 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 have 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. 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. The principal parameters of materials used in shot peening process are specified in Table 1. The materials used in shot peening process have been subjected to SEM analysis by means of microscope Ultra Plus (Zeiss, Germany) and microphotographs are presented on Fig. 1. The products obtained directly after sintering (without surface modification) have been used as reference specimens.

The surface roughness after shot peening process has been characterized by means of 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 × 5 mm using VSI method (Vertical Scanning Interferometry) and obtained signals have been converted by means of Bruker Vision64 software.

The next research phase encompasses the measurements of microhardness of modified surfaces. The measurements (20 indentations for each group of specimens) have been carried out under load of 0.2, 0.3 and 0.5 kg (HV0.2, HV0.3 and HV0.5 correspondingly) by means of Vickers FM-700 microhardness

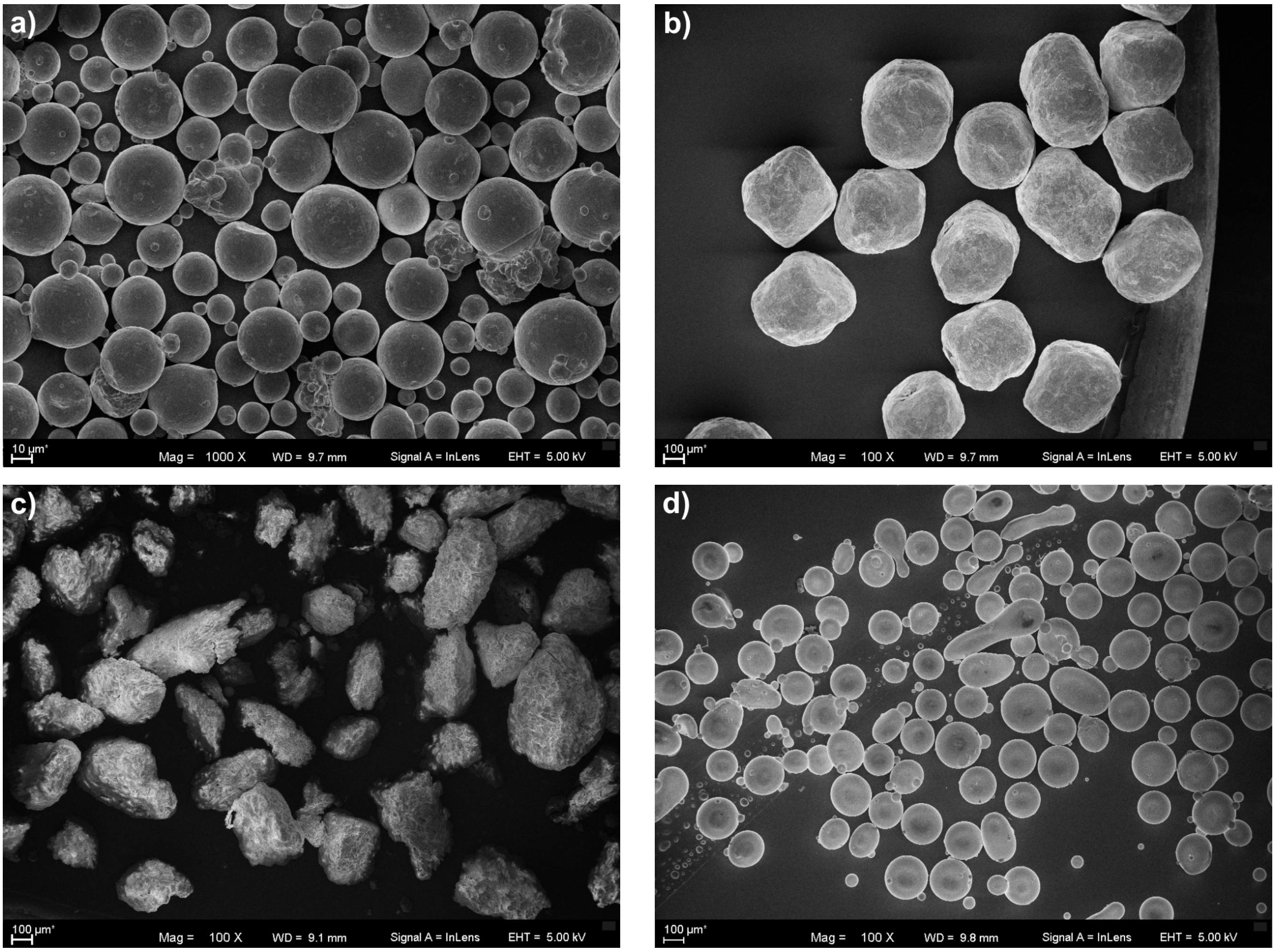

Fig. 1. SEM micrograph of: a) Ti-6Al-4V powder, b) stainless steel shot, c) nutshell granules, d) ceramic beads

TABLE 1

Parameters of shot for shot peening

Shot	Typical chemical composition (%)		Average grain size (µm)	Grain shape	Hardness
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

meter with automatic ARS 900 system (Future-Tech Corp., Japan). Wear tests have been carried out by means of ball-on-disc tribotester (CSM Instruments, Switzerland) in Ringer solution (pH = 7.2) at temperature of 37ºC. The balls with diameter of 6 mm made of Al_2O_3 with the hardness of 2000HV (CSM Instruments) have been used as counter-bodies (ball). The tests were carried out under load of 10N with linear velocity of 1.88 cm/s on the radius of 3 mm. The total test travel was equal to 100 m in course of which the variation of friction coefficient was recorded. The volumetric loss of specimen occurred as abrasion mark as a result of specimen and counter-bodies mating and has been applied as the measure of wear. Therefore, Dektak 150 contact profilometer (Veeco Instruments, USA) has been used on the specimen circumference (in 15 points) in order to measure the specimen abrasion profile area. The tip radius of measuring needle was equal to 2 µm. The volumetric wear has been determined as average value of the specimen abrasion profile area multiplied by abrasion mark circumference occurred in ball-on-disc test. Then so called wear coefficient K has been determined considering, except of volumetric wear, the load and the distance passed in course of test:

$$K = \frac{Wear\ volume}{Applied\ force \times sliding\ distance}\ [mm^3N^{-1}m^{-1}] \quad (1)$$

The surface of wear tracks in examined materials after executed tribological tests has been evaluated by means of scanning electron microscope Nova NanoSEM 450 (FEI, Holland).

3. Results and discussion

3.1. Morphology and geometrical structure of surface

The sintering process by DMLS technology takes place along the laser beam scanning direction on the surface of thin layer of powder deposited on the base plate. Therefore, as a result of surface tension on 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 [11] but excessive penetration of keyhole is unfavourable, because such phenomenon can generate the structural discontinuities i.e. pores in the product. Furthermore, the collapsing of welding puddle takes place in surface layer of the product (Fig. 2a). However, in accordance with information of Thijs et al. [14] gas bubbles in material can be generated additionally. Moreover, in the opinion of Yadroitsev et al. [15] the penetration significantly deeper than the thickness of sintered layer is also undesirable for energy reasons.

In course of SEM analysis of outer surface in X-Y plane (Fig. 2b) limited number of areas with not wholly melted metal powder grains has been found. In case of surfaces obtained directly after laser sintering process, structural discontinuities in the form of pores can influence as so called micro-notches. In real conditions existing in human body, they can constitute

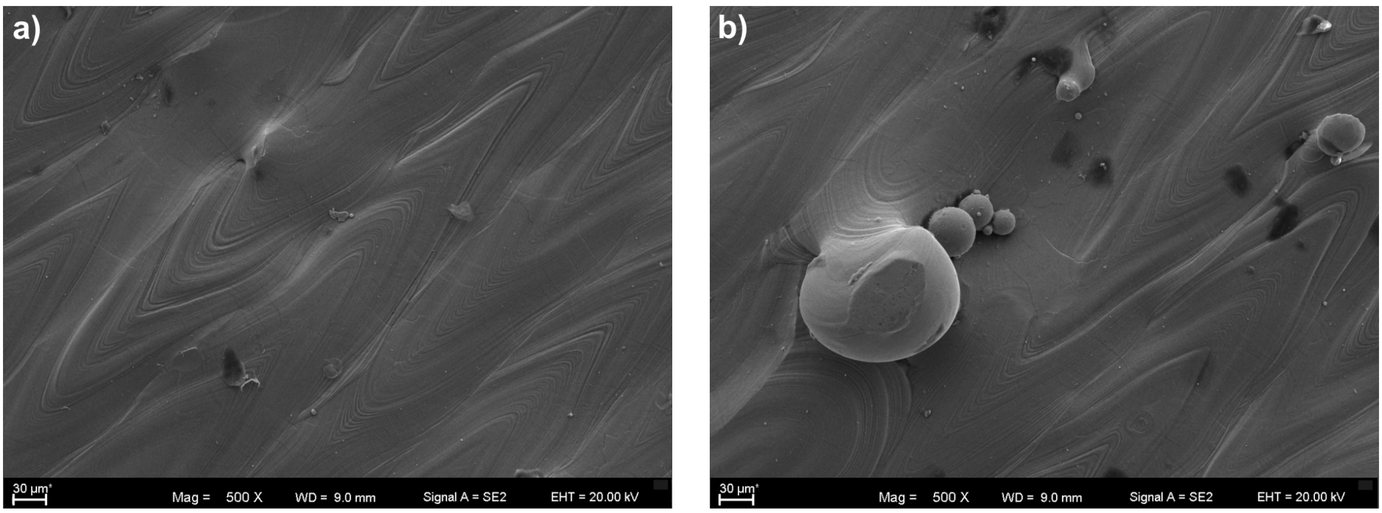

Fig. 2. SEM microphotographs illustrating the morphology of specimens surface after sintering by means of DMSL technology: a) visible laser beam scanning directions with structural discontinuity, b) partially melted titanium powder grains

the areas of micro-cracks development in surface layer under the influence of applied variable load and in direct vicinity of aggressive corrosion environment in the form of body fluids. Finally, such situation can cause accelerated release of metal ions and lead to the reduction of fatigue strength of an implant. Therefore, the use of shot peening process for metal products after 3D printing is additionally justified.

In case of SEM analysis of the surface after shot peening process (Fig. 3), we have observed shot grains in surface layer which are deposited in structural discontinuities or directly on the surface as a result of high levels of their kinetic energy. Ti-6Al-4V alloys belong to the group of materials characterized by significant ductility and plasticity as well as high reactivity which additionally contributes to shot penetration into surface layer. Penetrating shot can change geometrical structure, hardness and resistance to abrasive wear.

Metallographic observations of specimens cross-section (Fig. 4) after shot peening process using the highest pressure (0.4 MPa) indicated significant changes near the surface. The clear difference has been observed in the size of grains of the surface layer and the bulk substrate. There is significant reduction of grain size in surface layer and changes in microstructure reach to the depth of about 50 μm for substrate subjected to shot peening process by means of steel shot, about 25 μm for surface modified by means of nutshell granules and 60 μm for surface treated by means of ceramic beads. The changes in surface layer associated with the reduction of grain size have been also observed by Ahmed et al. [10] and Dai et al. [16] at different parameters of shot peening process. Moreover Kameyama and Komotori [12] observed that microstructure near the surface can additionally exhibit lamellar features associated with the transfer of shot particles fragments. In their study, they presented a model of lamellar microstructure created locally under the influence of fine particle peening.

Roughness analysis of the surface after shot peening process has been executed on the basis of *Sq* parameter (quadratic mean deviation). *Sq* parameter is statistically equal to standard deviation of profile ordinates and single high peak and valley of the profile more affect its value than *Sa* (arithmetic mean deviation). The results obtained from profilometric measurements are presented in Table 2. The correlation between *Sq* parameter and the value of shoot peening pressure is clearly visible i.e. the roughness increases with the increase of working pressure for all surfaces being modified. The amount of shot hitting the surface being processed is increased at higher pressure. In case of surface

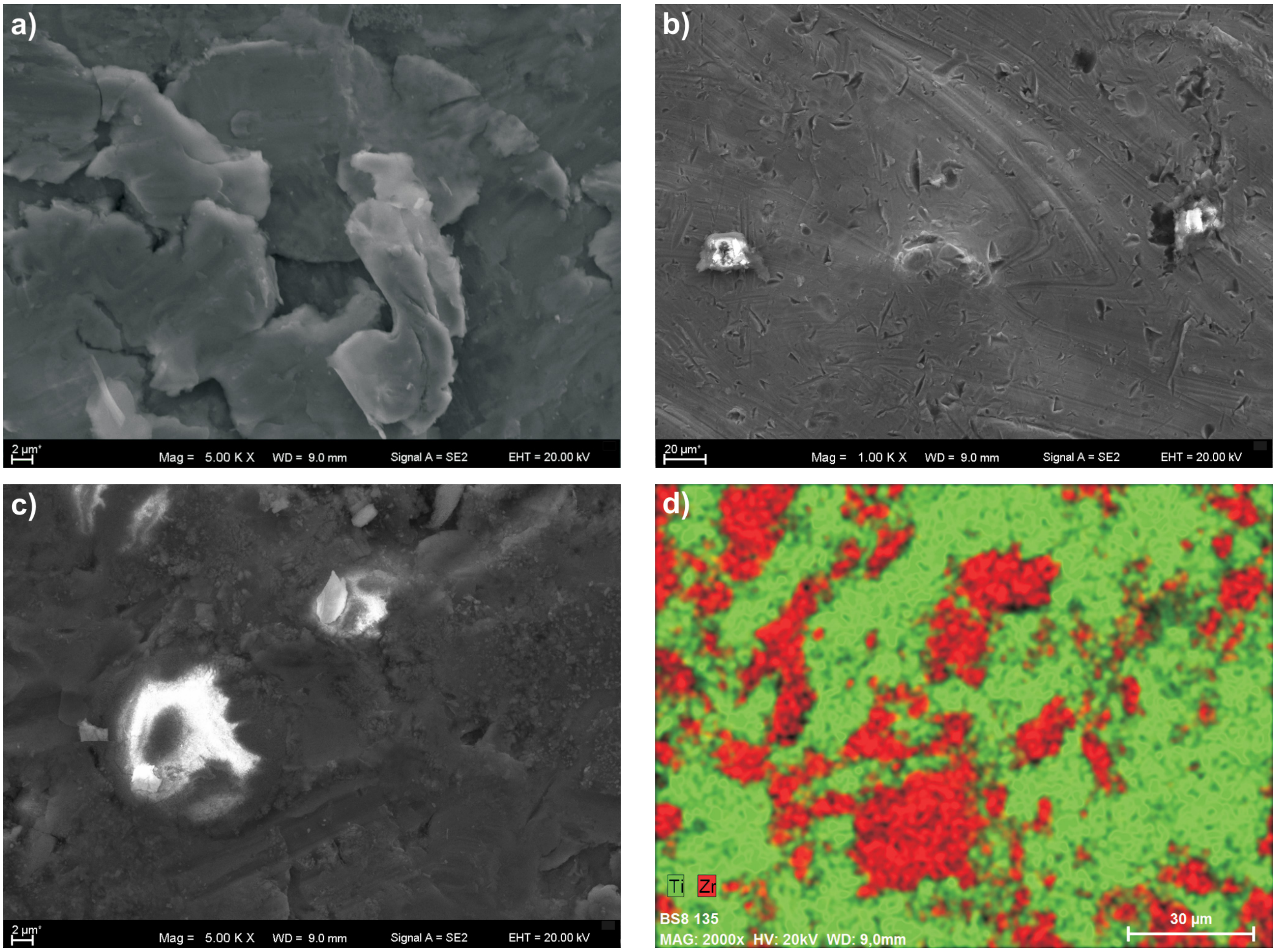

Fig. 3. SEM microphotographs illustrating shot inclusions on specimen surface: a) shot made of CrNi steel, b) nutshell granules c) ceramic beads d) Zr mapping originating from ceramic beads on background of Ti

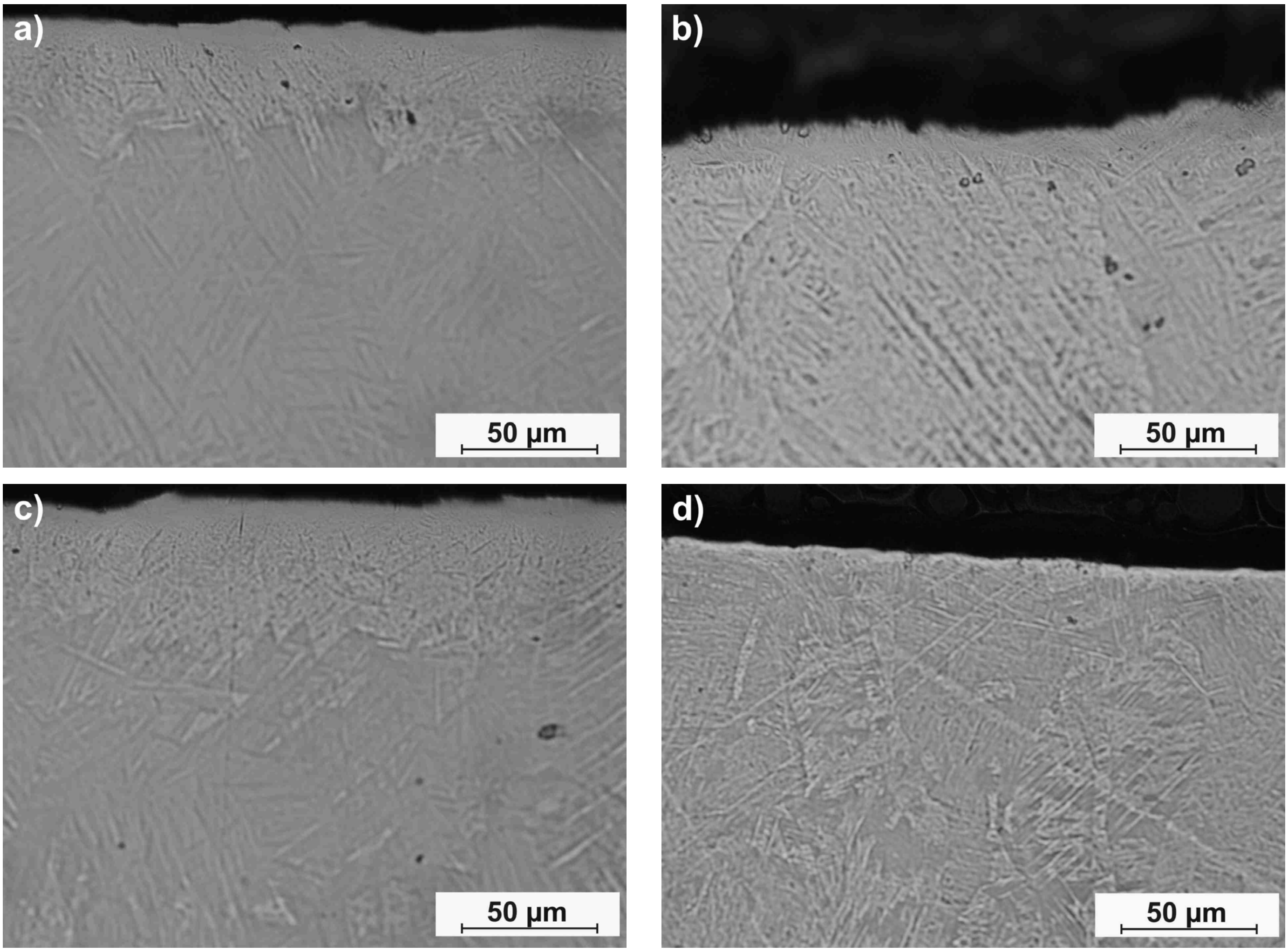

Fig. 4. The cross-section of specimens showing modified surface layer after shot peening using the pressure of 0.4 MPa: a) shot made of CrNi steel, b) nutshell granules, c) ceramic beads, d) reference sample

modification by means of steel shot or ceramic beads the degree of surface development is lower in comparison with the surface obtained directly after DMLS process. However in case of surface treatment by means of nutshells, the roughness is increased in comparison with reference specimen. Such situation is strongly associated with sharp edged shapes of nutshell granules while the shapes of CrNi shot and ceramic beads were almost spherical. The lowest *Sq* parameters have been obtained for surfaces treated with steel shot. Although, it should be emphasized that the values of *Sq* parameter for surfaces modified by means of CrNi shot have been obtained at average size almost three times larger than the size of ceramic beads. Smaller shot size leads to reduced size of indentation after peening but it is translated into increased number of indentations per unit of surface.

TABLE 2

Summary of quadratic mean deviation *Sq* (µm) for surfaces being tested

Peening pressure (MPa)	Steel CrNi	Nutshell granules	Ceramics	Unmodified surface after DMLS
0.2	6.423	9.744	7.804	
0.3	8.214	10.206	8.384	9.731
0.4	8.756	10.341	8.979	

3.2. Surface hardness

Microhardness tests were carried out in order to obtain information concerning the influence of shot peening process on mechanical properties of the surface. Fig. 5 illustrates the surface hardness vs. load. Comparing the obtained results, it can be noted that the hardness of specimens subjected to shot peening process is higher in comparison with untreated specimen. Furthermore, the increase of shot peening pressure causes the increase of hardness of the surface being treated. Obviously, the average hardness of specimen subjected to shot peening process by means of steel shot is the highest 468±29HV0.2; by means nutshell granules 366±21HV0.2, and by means of ceramic beads 454±11HV0.2. Therefore, the results obtained for the treatment by means of steel shot and by means of ceramic beads are extremely comparable. However, we have to remember that the values of microhardness of surfaces treated by means of

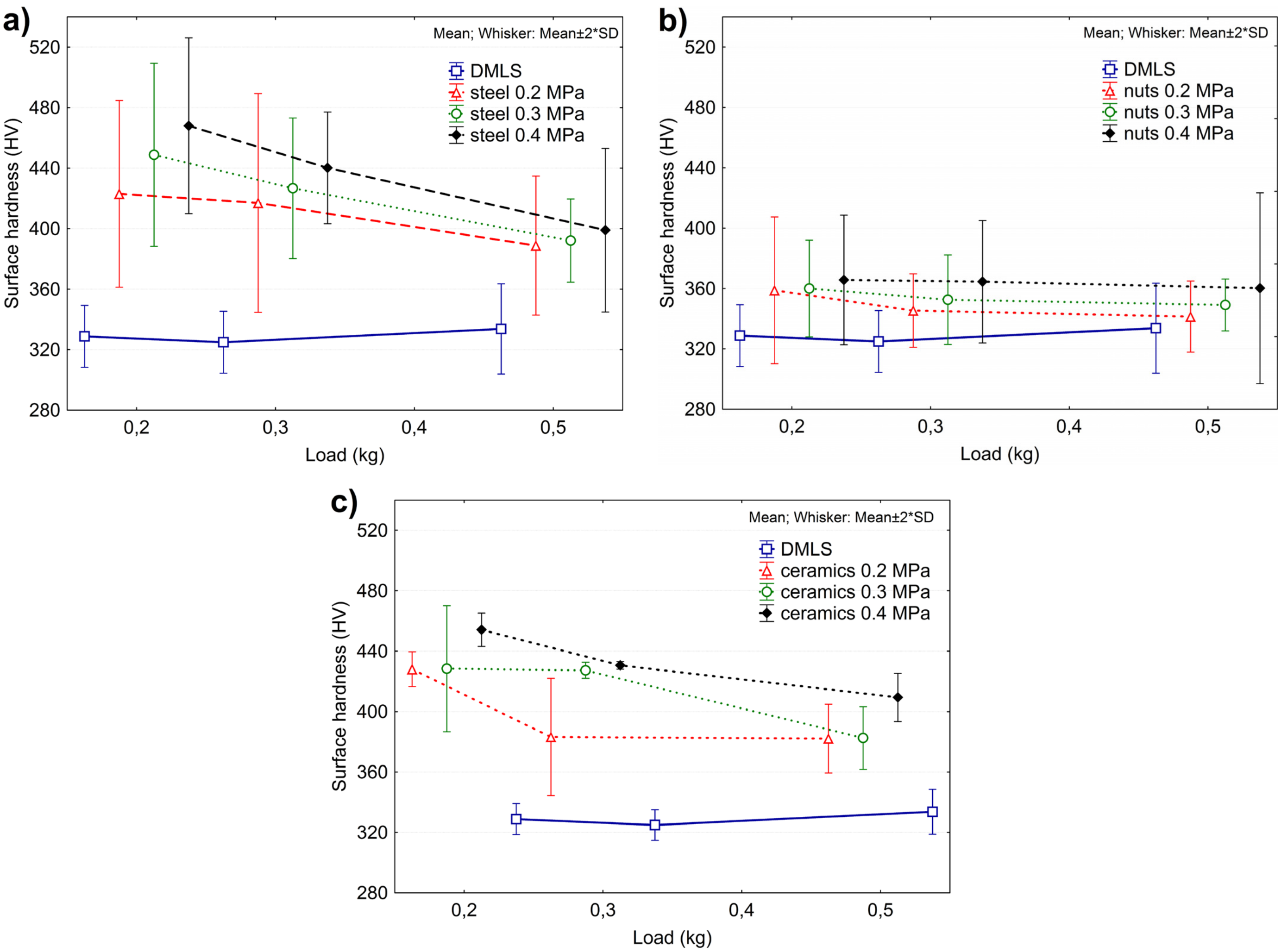

Fig. 5. The variation of hardness of Ti-6Al-4V alloy at different applied loads for shot peening surface: a) stainless steel shot, b) nutshell granules, c) ceramic beads

ceramic beads have been obtained by means of more than three times smaller size of shot. However, the maximum hardness of specimen not subjected to shot peening process is equal to about 329±10HV0.2 i.e. is almost equal to the value declared by powder manufacturer EOS GmbH – 320HV. The increase of surface hardness can be explained by the fact that nanocrystalline layer is created after shot peening process on the surface of specimens being treated which leads to material strengthening and to the increase of corrosion resistance [4,12]. As a result of plastic deformations in surface layer, hard shot residues are pressed in (penetrated). Furthermore, in the opinion of Ahmed et al. [10], titanium strengthening is possible to the depth of 0.1÷0.8 mm depending on shot peening parameter which explains the drop of average values of hardness vs. load increase (from 0.2 to 0.5 kg).

3.3. Wear and surface morphologies after ball-on-disc tests

The values of recorded friction coefficients are presented in Table 3 and graphical interpretation vs. distance is illustrated in Fig. 6. Comparative analysis demonstrated that the lowest values (but extremely similar) of friction coefficients have been recorded for surfaces treated by means of steel shot and ceramic beads. It has been observed that average friction coefficient decreases with increasing shot peening pressure for these two types of surfaces. Such behaviour of surfaces being tested is strongly correlated with the degree of surface layer strengthening i.e. hardness. The surfaces with the highest values of hardness were characterized by the lowest friction coefficient assuming that the roughness of surface layer is lower than the roughness of unmodified surface (reference specimen - DMLS). However in case of surfaces modified by means of nutshell granules, higher average friction coefficient has been obtained in case of Sq parameter higher than this parameter obtained for reference specimen. Additionally, from the curve illustrating coefficient of friction vs. distance on Fig. 6 it appears that the stability of friction coefficient is the highest in course of initial 25÷30 m where the impact of pressure on its value is particularly visible. Probably after the distance of 30 m, the counter – specimen made of Al_2O_3 is located out of strengthened material zone and changes (oscillations) of friction coefficient are more intensive at increased contact area between mating surfaces and penetrating products of secondary wear.

TABLE 3

Summary of friction coefficients determined for tested surfaces

Modified surface of sample	Peening pressure (MPa)	Average friction coefficients μ	Standard deviation
Steel CrNi	0.2	0.348	0.039
	0.3	0.345	0.035
	0.4	0.329	0.032
Nutshell granules	0.2	0.351	0.039
	0.3	0.372	0.042
	0.4	0.374	0.038
Ceramics	0.2	0.349	0.036
	0.3	0.331	0.037
	0.4	0.296	0.036
Unmodified surface after DMLS	—	0.358	0.043

Fig. 7a represents the results of friction coefficient for surfaces subjected to shot peening process. The highest wear resistance has been observed in Ringer environment for surfaces modified by means of ceramic beads. Considering min-max values and standard deviation, obtained values of wear coefficient K are comparable for surfaces modified by means of steel shot and ceramic beads. Definitely highest wear has been recorded for unmodified surface. Increased wear resistance can be observed for increasing shot peening pressure for surfaces modified by means of steel shot and ceramic beads. In this case, the increase of wear resistance is associated mainly with substrate hardness and with the depth of the strengthening as well as with the roughness lower than in case of surfaces treated by means of nutshell granules.

Opposite trend has been observed for surfaces modified by means of nutshells. Increased pressure of shot peening by means of sharp edged nutshells does not lead to high surface strengthening at relatively higher increase of Sq parameter. The surface layer abrasion process is mainly associated with the shearing of roughness peaks in initial test phase and intensified thereafter as a result of sliding along the surface with the potential presence of loose abrasive material (secondary wear products). In case of surfaces modified by means of nutshell granules, the depth of the strengthening is definitely lower than in case of other peening media. Small thickness of surface layer rich in nutshell fragments does not constitute any significant barrier for counterbody material as is the case in case of surfaces treated by means of surfaces treated by means of ceramic or steel shot. Therefore,

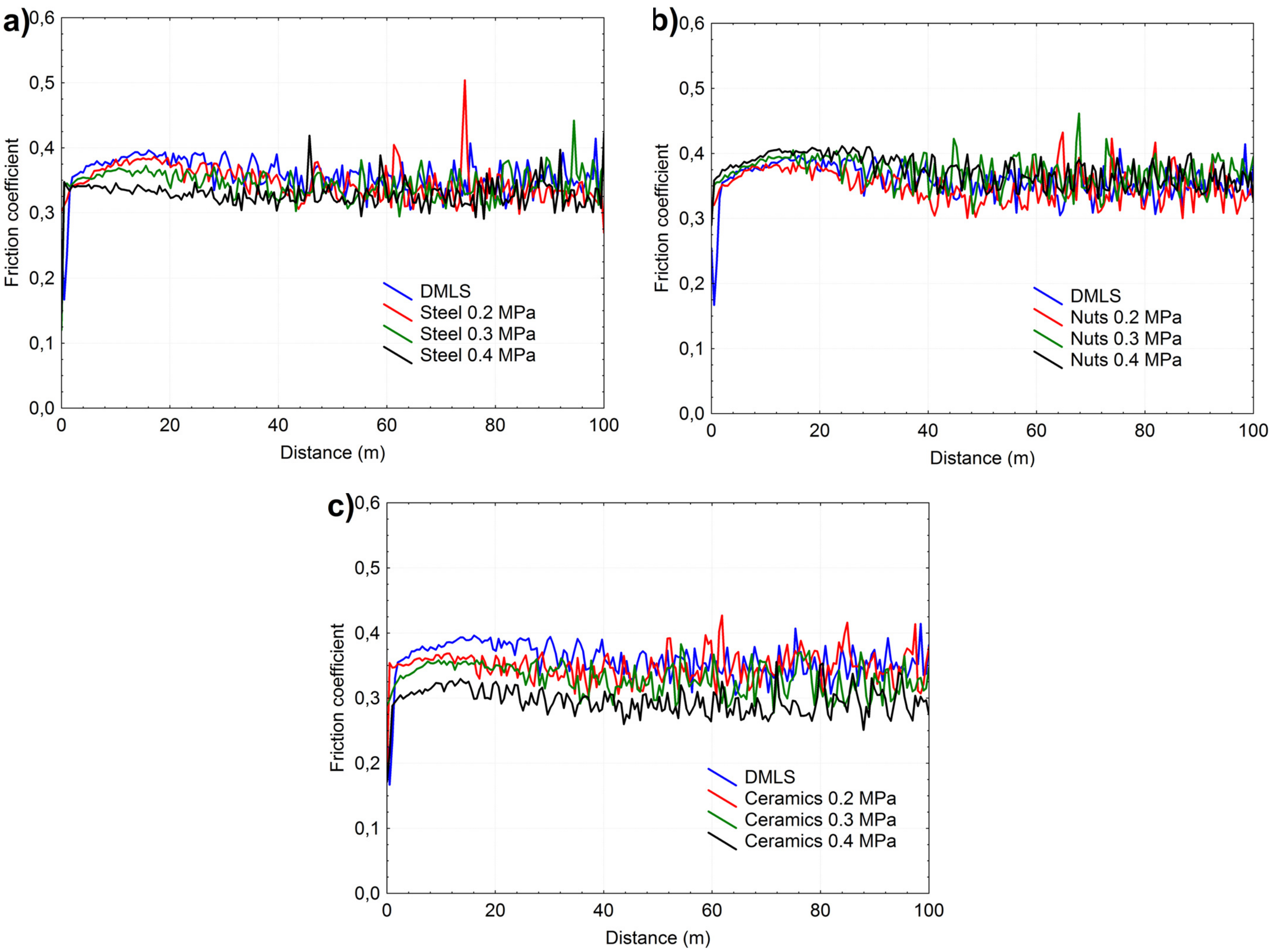

Fig. 6. The curve illustrating changes of friction coefficient vs. distance for surfaces modified by means of various methods: a) CrNi steel shot, b) nutshell granules, c) ceramic beads

in ball-on-disc test, the intensity of surface wear is associated with peaks shear process where the surfaces of a softer body will be smoothed as a result of abrasion or plastic deformations of roughness peaks.

Fig. 7b additionally illustrates the changes associated with the use of balls made of Al_2O_3 constituting the counter-body in the test. It has been observed that the wear of corundum balls was correlated with the hardness of substrate (disc) being tested. For relatively soft surfaces subjected to shot peening (at the lowest pressure) obtained values of wear factor were correspondingly lower. However, recorded differences between the groups of counter-bodies being tested are not statistically significant. Titanium alloy is characterized by high plasticity and low hardness against the high hardness of corundum ball (2000HV). Therefore, the wear factor of the balls is relatively low.

The surface of signs of wear after ball-on-disc tests has been subjected to SEM analysis (Fig. 8). In course of analysis it has been found that abrasive wear mechanism was prevailing on all surfaces subjected to analysis. Nevertheless, there are other parallel mechanisms, among others oxidizing process, occurring in all cases. However, this process was not as intensive as in the case of the tests carried out in technically dry friction conditions [17]. Generally, titanium alloys are characterized by high affinity to surrounding environment and passive layer is created spontaneously. According to the literature data [18], a passive layer about 1.5-5 nm can be created after just 1ns. Although the oxide layer is always present on titanium surface, this layer is extremely thin and weak. Therefore, it is easily broken and removed under the influence of counter-body and oxide debris is accumulated along wear paths valley. Such phenomenon was previously confirmed by Dong and Bell [17].

Furthermore, the signs of wear subjected to analysis are typical for metallic materials with significant hardness and high ductility e.g. Ti-6Al-4V alloy. A plastic zone with material accumulated along the direction of counter-body movement is visible. Prevailing wear mechanism consists in abrasion and groove forming. Such effect is caused by the presence of β phase (Ti-6Al-4V alloy consists of two phases $\alpha + \beta$). The plasticity of β phase is higher than the plasticity of α phase which contributes to locally increased plastic deformation. This phenomenon has been confirmed by research carried out by Faria et al. [19]. The phenomenon consisting in the creation of abrasive grooves parallel has been observed in all titanium alloy surfaces under analysis (Fig. 8a-d). However, the grooves forming observed on wear path was slightly more intensive (greater width of grooves) than in case of surfaces subjected to shot peening and the bottom of grooves was slightly smoother. Similarly, Bartolomeu et al. [20] indicate that, except of plastic deformation, it is abrasive grooves parallel that prevails in course of SEM observation of wear tracks.

Additionally, delaminations are observed along wear tracks (Fig. 8a-d) occurring as a result of multiple upsetting of the same material volume (low cycle fatigue phenomenon) caused by counter-body. Then, the wear process consists in the removal of successive material layers as a result of delamination of subsurface layer. Such mechanism occurs in course of sliding of solid bodies over each other with normal and tangential forces acting through adhesive and mechanical interaction in contact points. The surface of softer body is smoothed as a result of abrasion or plastic deformations of roughness peaks which leads to stress concentration in surface layer. The increase of surface layer and stresses leads to the occurrence of cracks in the subsurface layer which propagate under the influence of further loading and undergo deformations. Then a crack oriented toward the surface and material separation in the form "thin scale" take place in case of weakening of separated subsurface layer or local overloading.

Although, abrasive wear is the prevailing mechanism in case of alloy, adhesion phenomena associated with plastic deformation of secondary wear products (particularly visible on Fig. 8c) and material transport by counter – specimen are also possible (Fig. 8e). EDS analysis demonstrated that the chemical composition of counter – specimen surface (ball made of Al_2O_3) consists of mixture containing O, Ti, C, Al and small amount of Fe (probably residuals of steel shot). After the tests, the surfaces of all the alumina balls had the signs of slight single build-ups

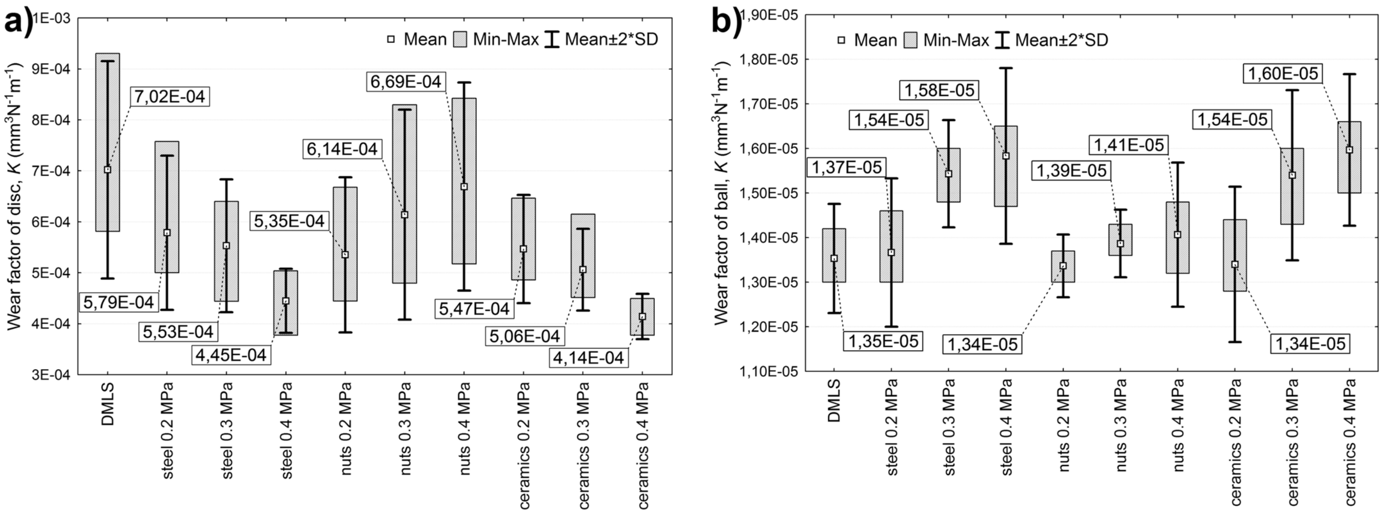

Fig. 7. Diagram illustrating wear factor K for tested materials: a) surfaces, b) counter-bodies

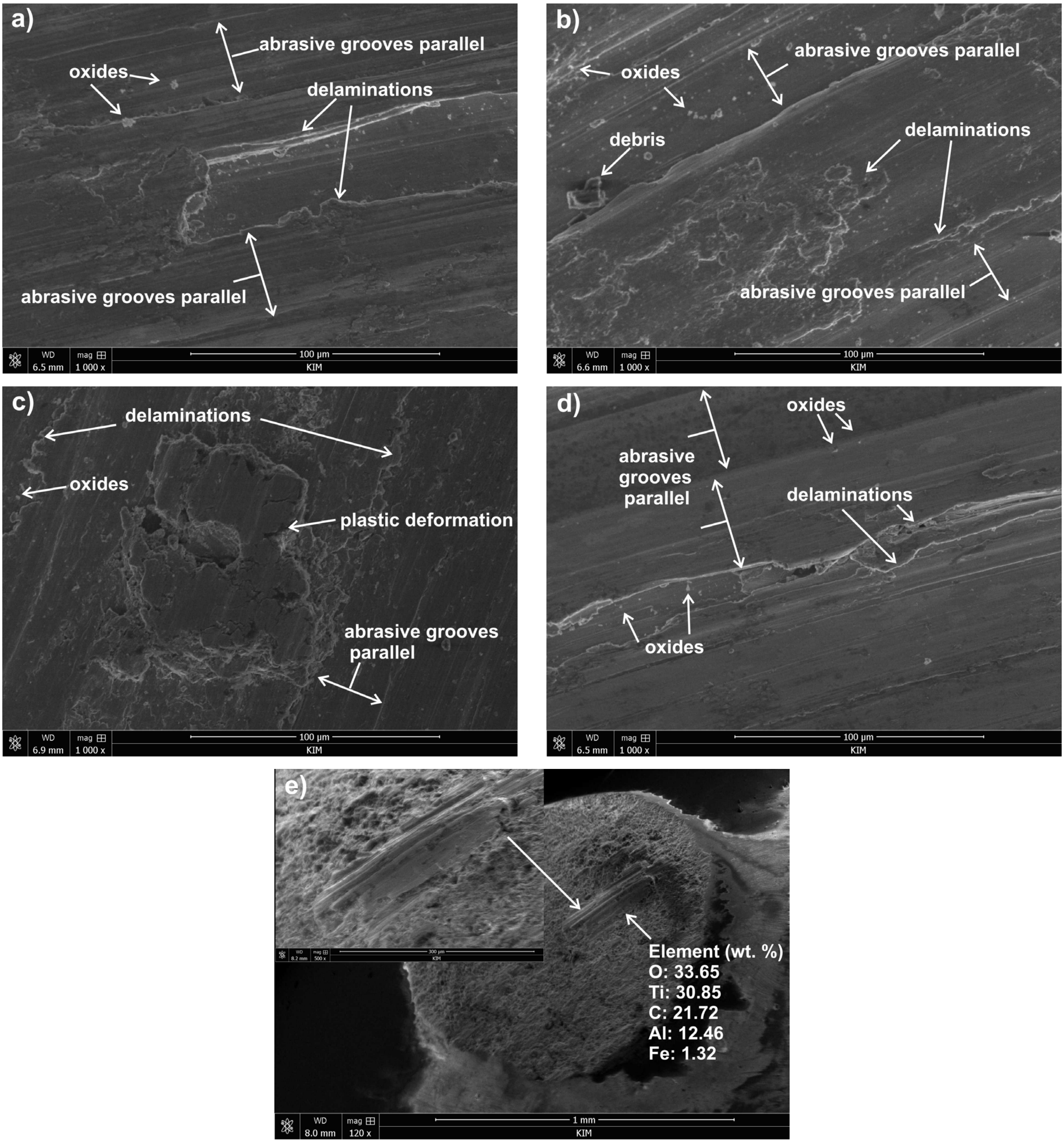

Fig. 8. SEM photographs of worn surfaces: a) modified by means of CrNi steel shot, b) modified by means of nutshell granules, c) modified by means of ceramic beads, d) reference sample (without shot peening), e) counter-body

indicating to material transfer. However, there were no significant differences in the size of adhering Ti6Al4V fragments. In course of wear tests in Phosphate Buffer Solution, Bartolomeu et al. [20] observed Ti-6Al-4V tribolayer adhering to alumina balls surface but the number of adhering particles was not as high as in case of tests carried out in technically dry friction conditions [17]. Moreover, in course of wear tests using steel counter-bodies, titanium transfer has been observed and confirmed by energy dispersive X-ray analysis.

4. Conclusions

The main conclusions that can be drawn from this study are:

– In course of SEM analysis of specimens obtained directly after DMLS sintering some discontinuities have been found with not wholly melted metal powder grains and pores. However, the presence of shot fragments has been found in case of specimens after shot peening process which leads to surface layer strengthening.

– Increased shot peeling pressure causes increased roughness of the surface while shot peening by means of medium with shape similar to sphere (CrNi steel and ceramics) causes the hardness reduction and the use of sharp edged nutshell granules leads to the hardness increased in comparison with reference surface.
– Shot peening by means of CrNi steel shot, nutshell granules and ceramic beads causes increased hardness of the surface being treated. Most favourable results have been obtained for surfaces modified by means of CrNi steel shot and ceramic beads under pressure of 0.4 MPa.
– Tribological tests in Ringer environment indicated the lowest values of friction coefficient and wear coefficient for surfaces modified by means of CrNi steel shot and ceramic beads. However, these values decrease with increasing shot peening pressure. The situation is opposite in case of surfaces treated by means of nutshell granules.
– Type of wear in the tested materials indicates to a typical abrasive wear mechanism, intensified by delaminations and groove forming. Furthermore, adhesion phenomena associated with material transport by counter-body are also observed.

The knowledge of tribological performance and condition of surface layer after materials shot peening will make it possible to develop the complex criteria for materials selection in course of medical products designing in future.

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