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Properties of the surface layer of titanium alloy Ti-6Al-4V produced by direct metal laser sintering technology after the shot peening treatment

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ABSTRACT

While considerable progress has already been made in the field of additive manufacturing further development is needed. There is a huge demand in the global market for the production of high-quality components with complex geometries. The post-production surfaces after fabrication by AM technologies require finishing treatment due to the presence of defects on the surface layer. Therefore, a series of studies have been carried out to analyse the effect of shot-peening treatment on DMLS-manufactured titanium specimens. The shot peening process was carried out using three different working media: CrNi steel shot, crushed nutshells, ceramic with working pressure of 0.3 MPa. The study included examination of surface roughness, Vickers hardness, phase composition and SEM analysis of the obtained surfaces. Analysis of the surface roughness showed a decrease in roughness using CrNi steel shot and ceramic balls. The use of nutshells resulted in an increase in roughness due to the sharp shape of the grains used. An increase in the surface hardness was observed for all modified surfaces. The least strengthening effect was obtained with nutshell shots. XRD phase analysis indicates that a two-phase structure of α'+β was identified in the post-production condition. There has been an increase in the share of the β phase for all treated samples. The greatest increase of the β phase was obtained for steel shots and ceramic balls. Changes in the percentages of the phases in the treated samples are the results of the induced phase transformations. The shot peening process also induces plastic deformation on the surface and forms a nanocrystalline layer, as demonstrated by SEM analysis. The overall results after the shot peening treatment indicates a favourable effect on the properties and the state of the surface layer of the Ti-6Al-4V titanium alloy for ceramic and steel shots.

Keywords: additive manufacturing, titanium alloys, DMLS, shot peening.

INTRODUCTION

Titanium alloys are known for their excellent physicochemical properties, such as good mechanical properties, biocompatibility and corrosion resistance at relatively low density [1, 2]. Their use, however, has expanded significantly as the cost of pure titanium has decreased by about 30% over the decade [3]. The main use of the titanium alloys is still in high-value industries such as aerospace, aeronautics or military application, but now it is also commonly used in fields as vast as in transport, energy, automotive, water treatment petrochemical industry and medicine [4].

For medical applications, the Ti-6Al-4V alloy is predominant [5]. The Ti-6Al-4V implants can be produced by conventional processes in the form of forging, casting, plastic forming [6, 7]. These methods produce a lot of material waste and often need extensive machining to final shapes and dimensions which results in high costs of this processes [8, 9]. The response to this high demand for patient-specific shaped products at low-cost and minimal material waste is the use of additive manufacturing technologies [10].

In additive manufacturing of titanium components, powder bed fusion (PBF) technologies play a leading role. Among them, technologies such as selective laser melting (SLM) and direct metal laser sintering (DMLS) stand out. Direct energy deposition (DED) technologies, including laser engineered net shaping (LENS) technology, also find application [11].

However, the structure of the surface layer of additively manufactured (AM) Ti-6Al-4V products are not ready for applications as components produced by these technologies are not defects-free and require further processing [12]. The main defects are in the form of porosities due to pool weld collapse, unmelted or semi-melted particles, as well as anisotropic properties [13]. Deposition of PVD nitride coatings is a possibility, with consideration of AlTiN and TiAlN thin films on the Ti-6Al-4V substrate [14].

However, the more popular technology for improving the state of the AM surface layer various are peening methods. There are many methods available for peening with unique and significant benefits. The peening methods include [15–17]:
- hot peening (SP),
- ultrasonic shot peening (USP),
- impulse shot peening,
- laser shock peening (LSP),
- water jet peening (WJP),
- oil jet peening,
- cavitation peening (CP).

The applications benefits of these surface modification technologies include enhanced mechanical properties in form of work-hardened layer with increase tribological wear resistance [18], improvement of corrosion resistance [19] and fatigue properties.

There are recent reports which indicate that using SP or LSP on either conventional [20] or 3D-printed [21] Ti-6Al-4V titanium alloy could be profitable on fatigue behaviour. An improvement in fatigue properties was also observed for CP [22] and USP [23]. Increases in safe fatigue life for titanium alloys are ranging from 25% [24] to as much as 200% [25]. The multitude of applicable processes, and their differential impact, results in the need to create and use precise models that determine the relationship between fatigue characteristics and total strain energy density as well as fracture surface topography parameters. One such model, which shows better accordance with fatigue test results compared to existing models based on the strain energy density, was presented by Macek [26].

It should be borne in mind that certain methods have their limitations and are not effective for specific manufacturing methods or modifications of titanium alloys. The recommended method for laser sintered products is the usage of SP treatment. This is related to the creation of unfavourable residual stresses in the implant after the use of laser sintering technology. The literature indicates that the further effect of shot peening process is removal of unfavourable residual stress and creation of favourable compressive stresses [27]. The plastic strain from shot peening leads to nanocrystallization in the surface layer [28, 29]. The shot grains can also penetrate to the surface layer creating the lamellar microstructure of embedded fragments of the shots and releasing the elements which changes in the chemical composition of the surface layer [30].

Therefore, the purpose of the present paper was to examine the effect of shot peening process on the properties of surface layer of specimens made of the Ti-6Al-4V titanium alloy produced by Direct Metal Laser Sintering (DMLS) process.

SUBJECT OF THE STUDY

Samples of Ti-6AL-4V alloy powder with a chemical composition according to ASTM F136 were made by DMLS technology using the EOSINT M280 metal powder laser melting system. After 3D-printing, the samples were shaped like disks with dimensions of 30 mm in diameter and 6 mm in thickness (Fig. 1a). The chemical composition of Ti-6Al-4V powder used in additive manufacturing is shown in Table 1. SEM image (Fig. 1b) exposed defects in the form of unmelted or semi-melted powder particles along with satellites and impurities on the surface, as well as voids after collapse of the welding pool.

Afterwards, the surfaces of the prepared specimens were shot-peened using IEPCO's Peenmatic micro 750S device with the peening pressure of 0.3MPa, peening time of 60s and three different shots: steel shots, nutshell granules and ceramic beads. A commercial peening medium from Kuchmichel Abrasiv GmbH was used. It is used in

Table 1. Chemical composition of Ti6Al4V powder

Element	*Al*	*V*	*Fe*	*O*	*C*	*Ti*	Others
Mass %	5–6.75	3.5–4.5	≤ 0.25	0.14–0.16	≤ 0.02	Bal.	0.4

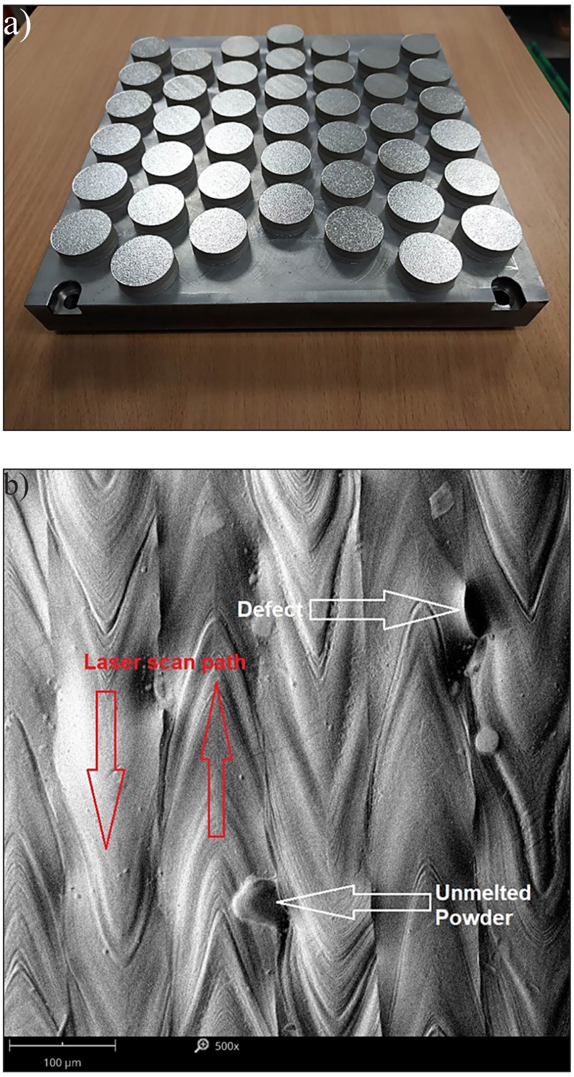

Figure 1. (a) The Ti-6Al-4V specimens after DMLS, (b) specimens microstructure as-built state

cleaning, preparation or surface modification processes. The characteristics of this shots, as stated by the manufacturer, are shown in Table 2 and SEM micrographs of the shots are shown in Figure 2.

RESULTS OF THE STUDIES

SEM and EDS analysis of the surface after shot-peening

The surface of the samples was evaluated using a Phenom ProX scanning microscope, from Phenom-World. The study was carried out at 500× magnification, using the BSD backscattered electron detector and Topo A topographic mode, as shown in Figures 3–5.

Observations on the surface directly after 3D printing show some kind of structural discontinuities in the form of unmelted or semi-melted metal powders, and collapsed weld pool marks which can be seen on passing laser paths. In the case of the peened surfaces, plastic deformations formed after the shot peening treatment were found. According to the literature [30], each shot impact on the surface causes the shot residue to embed itself in the material structure. This leads to the formation of a lamellar structure. Using the BSD observation mode, a difference in imaging can be observed – dark reflections represent nut shell residues and ceramic balls, while bright reflections indicate steel shot residues.

In addition, an EDS analysis was also performed, which showed changes in the chemical composition of the peened surface layer by releasing of elements and contribution to the formation of a lamellar nanocrystalline layer. There was an increase in oxides after CrNi steel (iron oxides) and ceramic shot peening (zirconium and silicon oxides), while there was an increase in carbides after nutshell peening. Analysis of the chemical composition is shown in Table 3 with the specification of the point studied in Figure 6. EDS spectra for all surfaces are shown in Figure 7. SEM-EDS analysis also showed residual shot located in

Table 2. Shot parameters of shot peening Ti-6Al-4V (Kuchmichel Abrasiv Gmb)

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

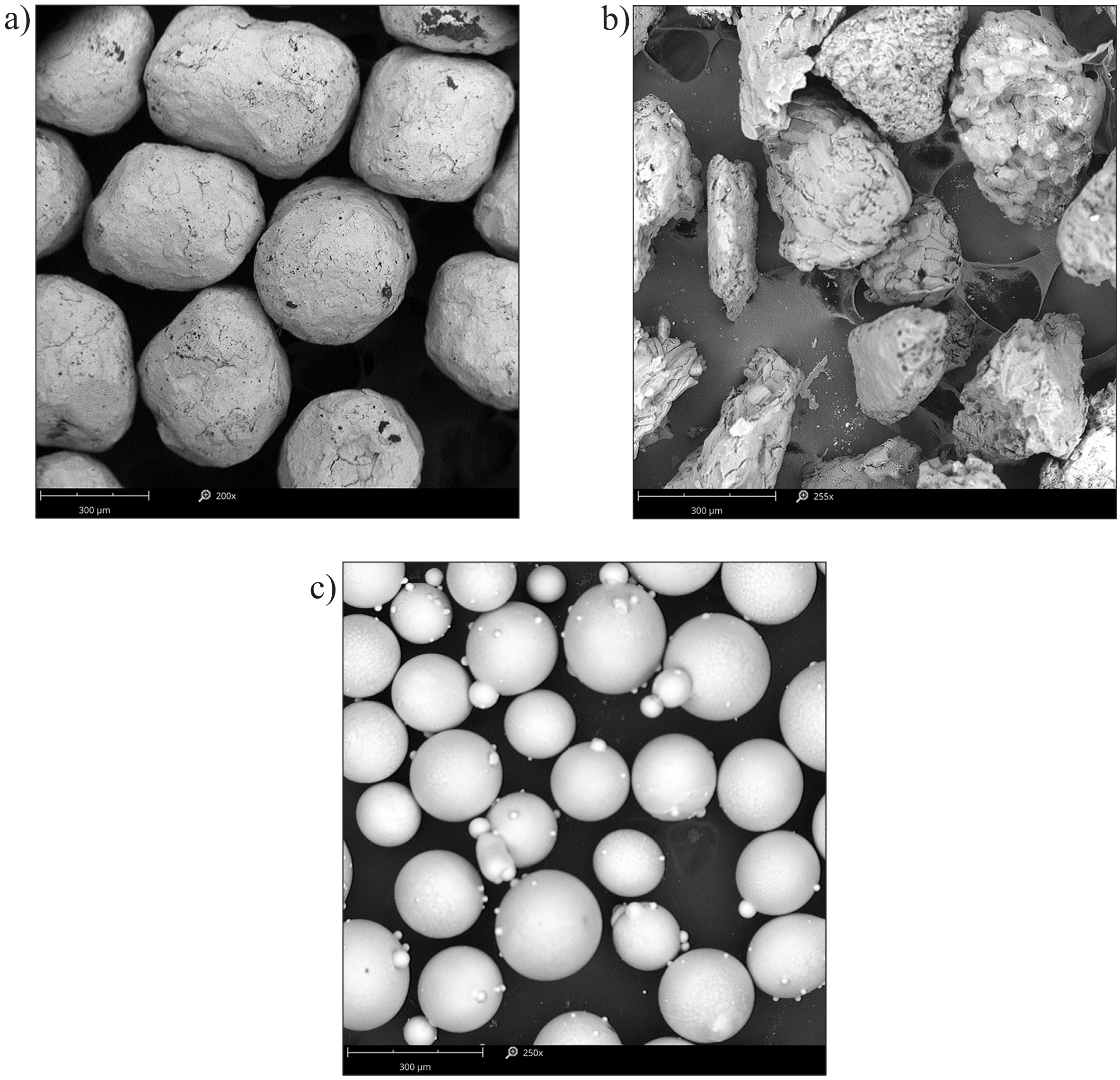

Figure 2. SEM micrograph of: (a) stainless steel shot, (b) nutshell granules, (c) ceramic beads

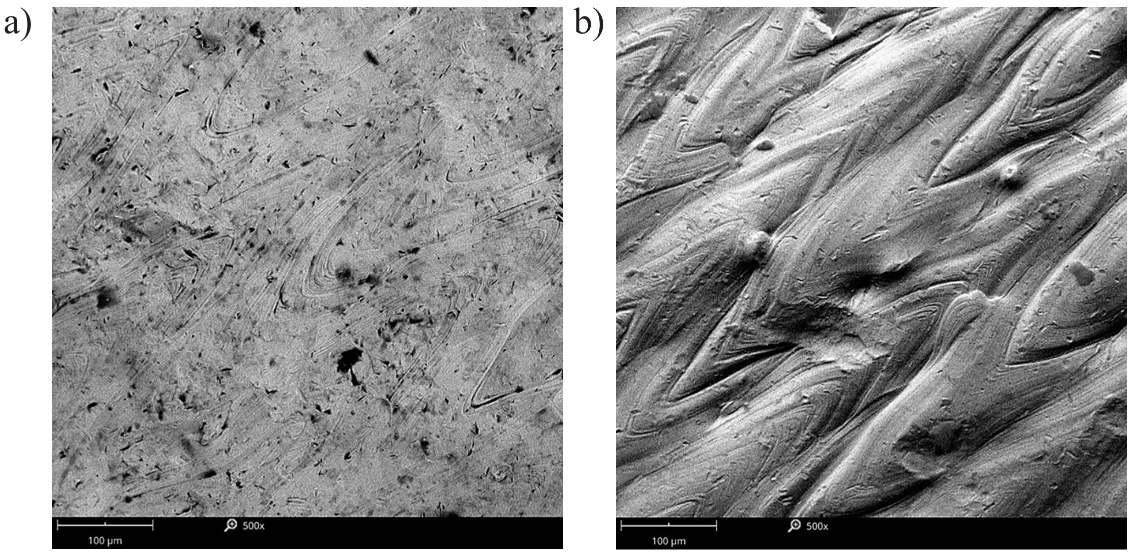

Figure 3. Surface of the Ti6Al4V sample after shot-peening with nutshell granules (a) with BSD detector, (b) in Topo A mode

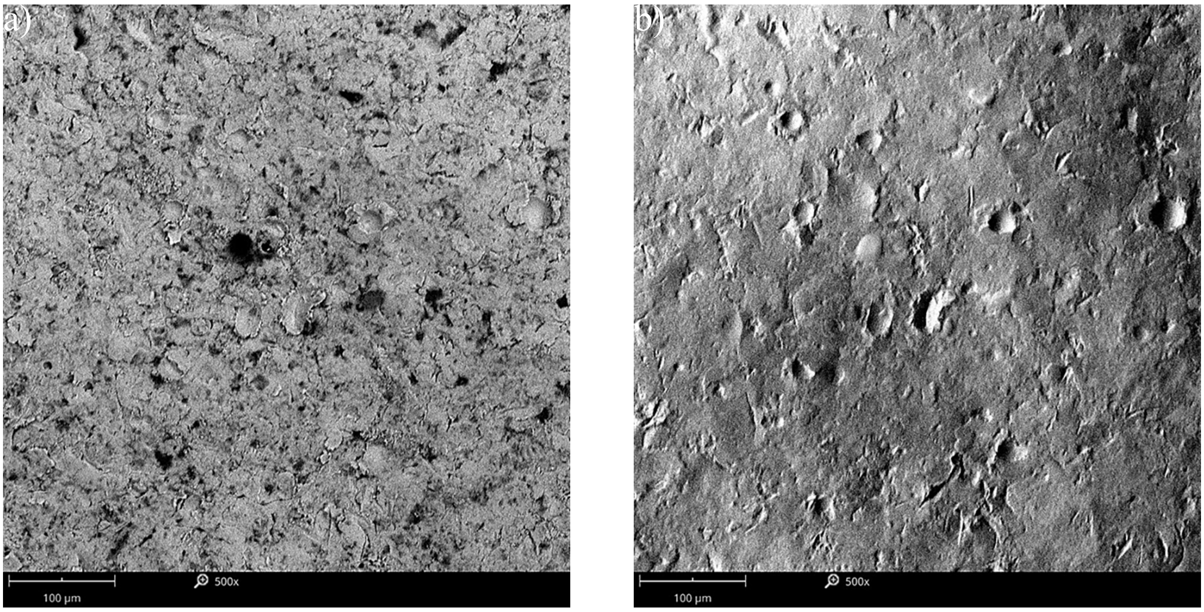

Figure 4. Surface of the Ti6Al4V sample after shot-peening with ceramic beads (a) with BSD detector, (b) in Topo A mode

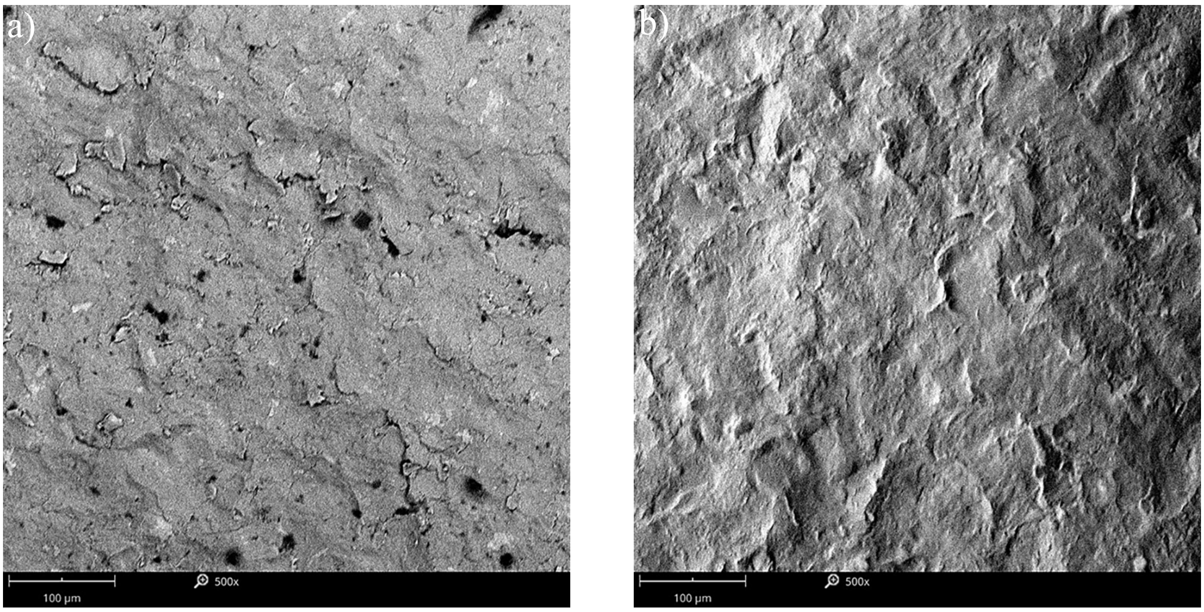

Figure 5. Surface of the Ti6Al4V sample after shot-peening with stainless steel shots a) with BSD detector, b) in Topo A mode

the surface layer of the material which may affect cytotoxicity. In the case of CrNi steel shot, there is a metallosis effect which affects the penetration of wear debris created in the course of prosthesis use, as well as osteolysis and aseptic loosening secondary to wear debris belong [31]. It is necessary to look for a medium with a negligible or inert effect on the organic environment and the results for ceramic beads and nut shells are promising under the application of cytotoxicity.

Phase composition analysis

Phase analysis was performed at room temperature using a high-resolution X-ray diffractometer (XRD, Empyrean, Panalytical) with Cu K-α radiation and a Ni filter, and a generator with a voltage of 40 kV and a current of 30 mA. The samples were measured in Bragg-Brentano geometry from 2θ = 30° to 100° with a step of 0.01° and a counting time of

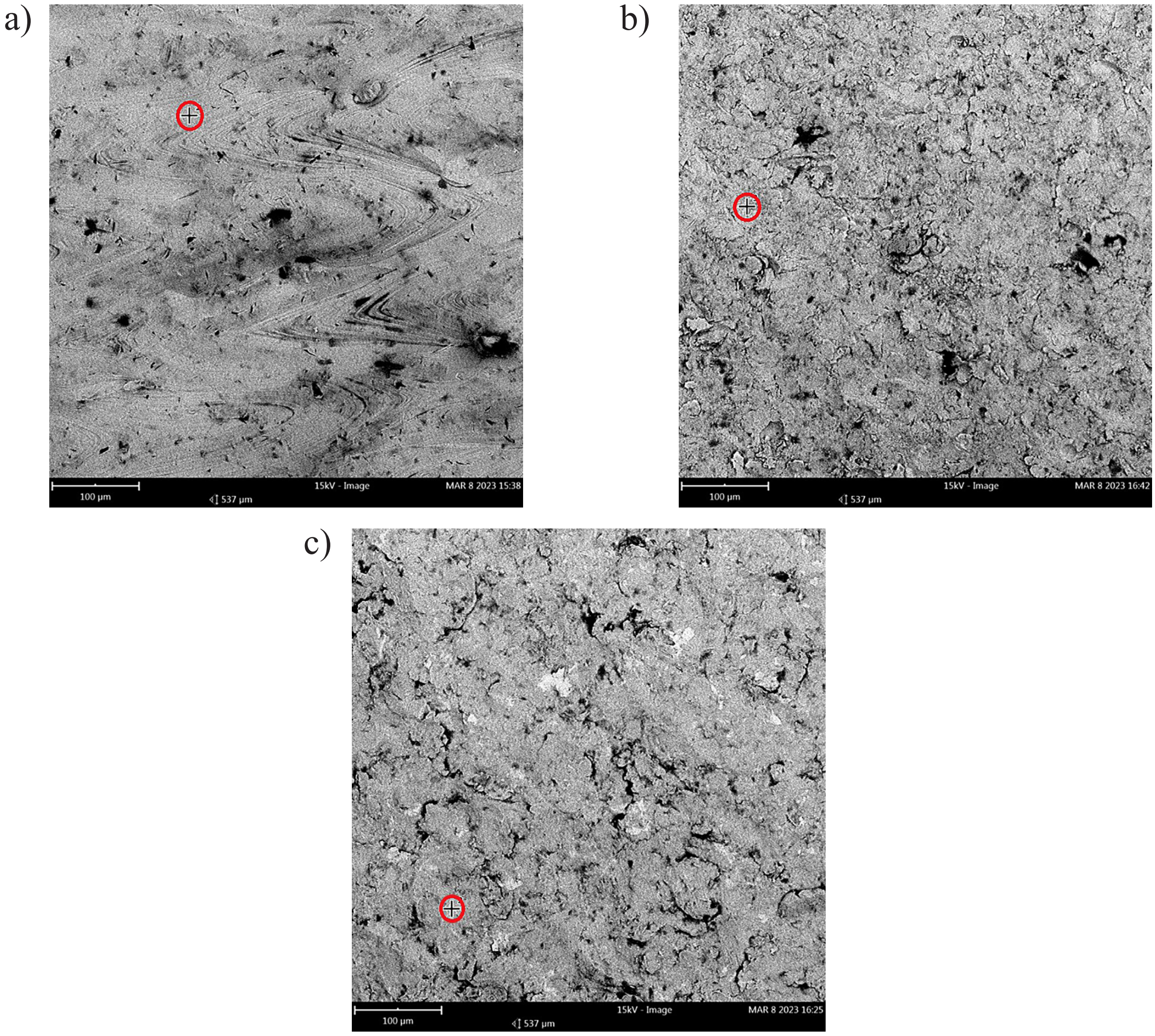

Figure 6. Surfaces of the Ti6Al4V sample after shot-peening with spot under EDS examination: (a) nutshell, (b) ceramic beads, (c) steel shots

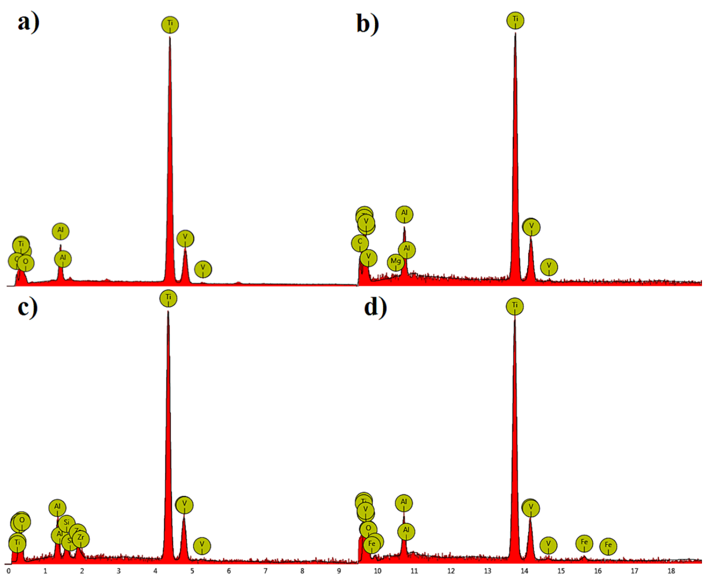

Figure 7. EDS spectra of: a) unpeened surface, b) surface peened with nutshell c) surface peened with ceramic balls d) surface peened with steel shots

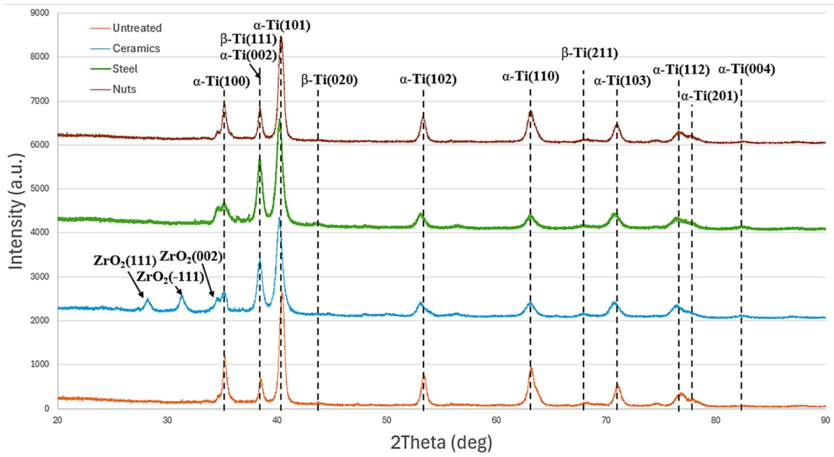

Figure 8. XRD patterns of Ti6Al4V titanium alloy before and after the shot peening process using ZrO_2 beads, CrNi shots and nutshell granules

6 s per data point. The results of the spectral analysis are shown in Figure 8 and the percentage of each phase were conducted by means of Rietveld analysis and summarized in Table 4. Phase analysis showed that a two-phase α'+β structure was identified in the as-prepared state. Changes in the percentages of the phases were found in the

Table 4. Proportion of individual phases in Ti-6Al-4V samples

Sample	α'-Ti (%)	β -Ti (%)
Untreated	85.0	15.0
Nutshell	80.0	20.0
Ceramic beads	63.0	37.0
Steel shots	61.0	39.0

Table 3. Comparison of chemical composition (% mass) of the Ti6Al4V powder to the composition of surfaces after shot peening treatment with different shots (nutshells, steel shots, ceramic balls)

Surface	*Al*	*V*	*Fe*	*O*	*C*	*Ti*	*Others*	
Untreated	5–6.75	3.5–4.5	≤ 0.25	0.14–0.16	≤ 0.02	Bal.	≤ 0.4	
Nutshell granules	7.15	3.49	-	-	16.30	72.86	0.21 (Mg)	
Stainless steel shot	5.64	2.80	2.77	23.16	-	65.63	*Zr*	*Si*
Ceramic beads	4.43	3.14	-	26.21	-	60.60	3.78	1.85

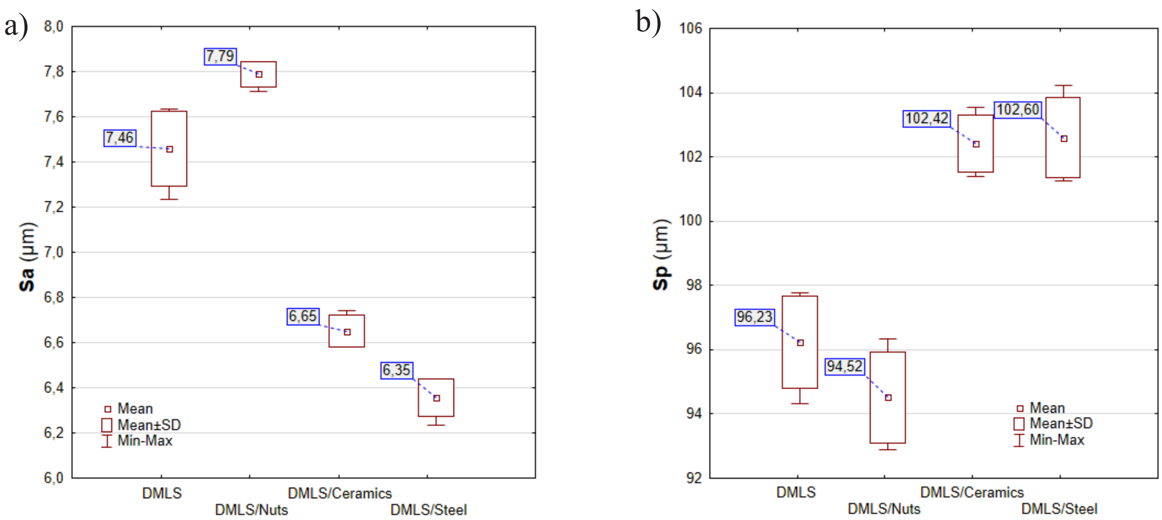

Figure 9. Results of roughness parameters measurements: (a) arithmetical mean height of the ordinates of the 3D profile, (b) maximum peak height of the 3D profile

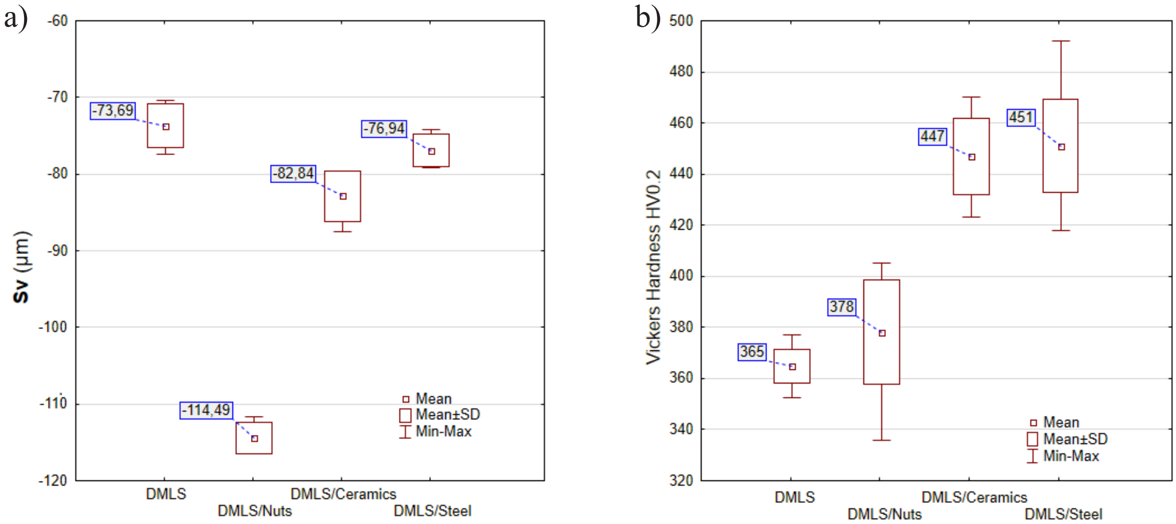

Figure 10. Results of roughness and hardness measurements: (a) maximum pit height of the 3D profile, (b) hardness results HV0.2

treated samples in the form of an increase in the proportion of the β phase due to induced phase transformations. A similar pattern of induced phase transformations was observed in the shot peening of 17-4PH steel after DMLS printing. The results are described in more detail in the paper [32].

Roughness and hardness measurements

Stereometric evaluation of the surface was performed on a Contour GT optical profilometer. A randomly selected area of 5×5 mm was analyzed. Three roughness parameters were determined: Sa (the arithmetic mean of the ordinates of the 3D profile), Sp (the height of the highest elevation of the 3D profile), Sv (the value of the lowest indentation of the 3D profile). Hardness tests, on the other hand, were carried out on the surface of the modified specimens by the Vickers method using an FM-800 hardness tester. Measurements were carried out for 10 seconds under HV0.2 load (200 mg, 1.96 N) in accordance with PN-EN ISO 6507-1. 20 hardness measurements were made. The results of the surface roughness and Vickers hardness are shown in a box-and-whisker plots in Figures 9–10

Surface modification with steel shot as well as ceramic balls results in a decrease in roughness parameters compared to the unmodified surface. In contrast, there is an increase in roughness when modified with nutshells. This is probably due to the shape of the peening medium. Steel and ceramic shots have a near spherical shape, compared to irregular and sharp-edged granules of nutshells. The results of the hardness test show a slight increase in the hardness value after shot peening with nutshells compared to unmodified samples. The increase averaged about 4%. A significant increase was recorded for the specimens treated with other medium, which amounted to about 23%. Crucial to the strengthening of the layer after SP treatment is the “crush” effect, the increase in dislocation density, the induced phase transformation shown by XRD analysis and the localized shot residues in the surface layer shown by SEM analysis. These factors were mentioned as influencing on a strengthening effect, among others, by Skoczylas [33] when comparing vibratory shot peening to conventional.

CONCLUSIONS

The overall results after the shot peening treatment indicates a favourable effect on the properties and the state of the surface layer of the additively manufactured Ti-6Al-4V titanium alloy. In terms of reducing roughness as well as increasing hardness, the best results were obtained for the surfaces peened with ceramics and steel shots. Equally satisfactory results were obtained for martensitic steel 17-4PH [34] and austenitic steel X5CrNi18-10 [35] used in the manufacture of medical devices. Therefore, it can be hoped that titanium products will also be widely used in the medical industry.

In similar studies [36] on the surface layer properties of titanium alloy Ti6Al4V, the phase changes and chemical composition of the surfaces studied were not taken into account, which is a novelty in the work presented here.

In this area, research is needed into obtaining optimum processing parameters to find a compromise between hardening and surface smoothness, taking into account the impact on the biological environment. Selecting the correct peening pressure is vital, as increasing pressure results in an increase in strengthening; however, it also results in a deterioration of surface smoothness, an increase in the amount of embedded shot residue, which should have a direct impact on cytotoxicity.

On the basis of the study, the following conclusions can be drawn:

- SEM images revealed that the shot-peening process causes plastic deformation on the surface, as well as shot residues in the microstructure which are imaged using the BSD technique in the form of light (nutshells) and dark reflections (steel and ceramic shot).
- SEM-EDS analysis showed the formation of numerous oxides and carbides, which contribute to the formation of a lamellar nanocrystalline layer on the surface of the treated surfaces.
- XRD analysis showed two-phase α’+β, as well as changes in the percentage of each phase by increasing the β phase after the peening process. The largest increase in the β-phase was observed for steel shots (from 15% to 39%).
- There has been a reduction in surface roughness (Sa) after shot-peening using Cr-Ni shot and ceramic balls, while the process leads to an increase when using nutshells.
- The highest peak height (Sp) was recorded for the specimens treated with ceramic beads and steel shots. The lowest pit height (Sv) was recorded for the specimens treated with nutshells.
- Vickers hardness analysis showed surface strengthening after shot-peening. The largest was obtained for ceramic as well as steel shot by about 23%. While the smallest for nutshells of about 4% indicating a cleaning effect when using this medium.

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