DOI: https://doi.org/10.37434/tpwj2022.09.04

INFLUENCE OF HEAT TREATMENT OF SPECIMENS FROM Ti6Al4V MANUFACTURED BY THE TECHNOLOGY OF SELECTIVE LASER MELTING ON STRUCTURE AND MECHANICAL PROPERTIES

S.V. Adzhamskyi[1], **G.A. Kononenko**[1,2], **V.R. Podolskyi**[1,2,3]

[1]LLC “Additive Laser Technology of Ukraine”
31 v Str., Serhiy Podolynskyi, 49000, Dnipro, Ukraine
[2]Z.I. Nekrasov Iron and Steel Institute of the NASU
1 Academician Starodubov Sq., 49000, Dnipro, Ukraine
[3]Ukrainian State University of Science and Technologies
4 Gagarina Prosp., 49000 Ukraine, Dnipro, Ukraine

ABSTRACT

Additive manufacturing, in particular, selective laser melting (SLM) is a modern method of manufacturing parts and assemblies of a complex geometry from metal powder, which are difficult or impossible to be reproduced in the states of traditional manufacturing. This technology is featured by residual stresses formed at the stage of manufacturing parts. Since titanium alloys are characterized by low thermal conductivity, the problem of forming residual stresses is of particular relevance for them and heat treatment for their removal is mandatory for products manufactured using SLM technology. Since the structural state of products manufactured by SLM technology differs from that formed with the use of traditional technologies, it is necessary to study the effect of annealing for residual stress removal on mechanical properties and microstructure of Ti6Al4V alloy manufactured by SLM technology. The specimens were studied after annealing with an exposure time of 1–5 h at 800 °C. It was found that as compared to the initial state after manufacturing, the ultimate strength after heat treatment for 1–5 h undergoes a decrease by 20.55– –23.03 % and relative elongation has an increase by 31.33–35.57 %. At the same time, the nature of variation in the values of relative reduction in area is non-uniform: annealing with an exposure of 1 h does not cause significant changes; with an increase in the exposure time to 2, 3, and 4 h, a decrease in this characteristic is observed, respectively, by 9.03; 45.97 and 62.56 % as compared to the initial state; after exposure for 5 h, the value of relative reduction in area undergoes an increase in this characteristic as compared to the values after exposure for 4 h — by ~26.12 %. According to the results of the correlation analysis of the values of mechanical properties and microstructure parameters, it was found that the shape factor of α-phase lamellas has a high ratio with the values of ultimate strength, and the amount of α-phase is most correlated with the values of relative reduction in area at static tension

KEYWORDS: selective laser melting, heat treatment, titanium Ti6Al4V alloy, mechanical properties, microstructure

INTRODUCTION

Technologies of additive manufacturing (AM), also known as 3D-printing, are increasingly used in the recent times, as well as a number of materials and methods that can be used is expanding.

Since manufacturing processes are ever improved and developed, a demand for more rapid and less expensive manufacturing processes provided the development of a number of rapid prototyping (RP) processes. Using additive manufacturing, almost any geometry with variations in size and complexity can be manufactured with a high degree of accuracy (Simchi & Asgharzadeh, 2004). RP technologies allow manufacturing parts with a complex three-dimensional geometry using AM.

By means of the process of selection laser melting (further — SLM-technology), specimens with a complex geometry from metal powder are manufactured, which are impossible or difficult to be made by other traditional methods of manufacturing [1].

One of the advantages of AM is only a small amount of further treatment (polishing, sandblasting, heat treatment) of products, thus expensive processes with the additional cost can be minimized [2]. Due to a significant widespread of AM technologies, the treatment of such materials has become of particular importance in recent years [3].

One of the features of SLM technology, according to a number of authors [1, 4–7], is the formation of defects that have a different nature as compared to the traditional method of manufacturing, and the possibilities of eliminating these defects at the stage of manufacturing process, or at the stage of further treatment. One of the main defects of additive manufacturing is residual stresses [5, 7], which are formed at the stage of manufacturing parts, their value depends on the parameters of the technological process, they can lead to distortion of the geometry (Figure 1, *a*), cracking

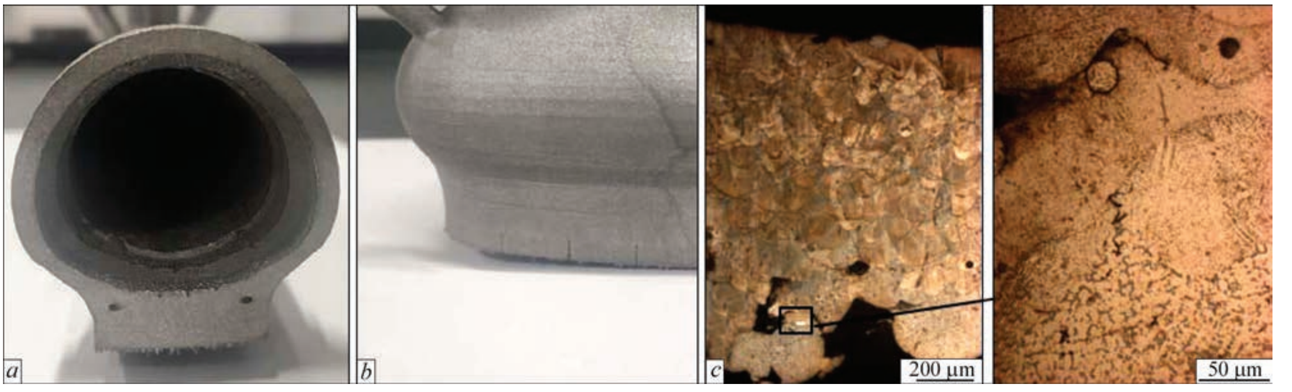

Figure 1. Defects of residual stresses: *a* — distortion of geometry; *b* — cracking; *c* — liquation cracking

of a 3D-product (Figure 1, *b*) and liquation cracking (Figure 1, *c*).

The occurrence of inner residual stresses in parts manufactured using SLM technology is associated with the process of very rapid solidification of the melt pool, several times recrystallization and a local coexistence of hot and cold metal. During cooling in the melt pool, tensile stresses occur as a result of solidification shrinkage and thermal compression during phase transformations in the process of crystallization. Under the action of these stresses, a system of microscopic compressive stresses can be formed, which can lead to cracking. As inner residual stress concentrators, also splashes of liquid alloy on the substrate in the process of powder scanning may be. As far as they have a lower temperature, as a consequence, a large temperature gradient and microcracking arise. Distortion of the geometry during the scanning process and a subsequent application of powder can lead to uneven application of the next layer of powder. Further, this will increase distortions, which provokes an emergency stop of the process of manufacturing parts by using SLM technology, since this technology has a tendency to accumulation of inner residual stresses.

The previous studies [5, 7], aimed at finding the ways of reducing inner stresses, allowed establishing, that the use of rational energy parameters of the powder scanning (beam movement speed and laser power) and a certain building strategy allow reducing deformations under the action of residual stresses without the loss of a product density.

Ti6Al4V alloy has been widely used in manufacturing products of different purpose, including those using AM due to the optimal combination of technological and mechanical properties. However, titanium alloys are characterized by low thermal conductivity, which makes the problem of residual stress formation even more relevant, and the heat treatment for their removal is mandatory for products manufactured using SLM technology. However, it should be noted that the structural state of products manufactured using SLM technology is different from the one that is formed using traditional technologies. Therefore, it is necessary to conduct studies of the impact of annealing for relief of residual stresses on mechanical properties and microstructure of Ti6Al4V alloy, manufactured using SLM technology.

The heat treatment of titanium alloys mostly includes only annealing for relief of residual stresses at 800 °C, which is carried out in an inert atmosphere to prevent surface oxidation. This work will determine the impact of different exposure time during heat treatment of experimental specimens of Ti6Al4V alloy on the mechanical properties during tensile tests.

The aim of the work is the determination and comparing of mechanical properties of tensile test specimens before and after the heat treatment at different exposure time.

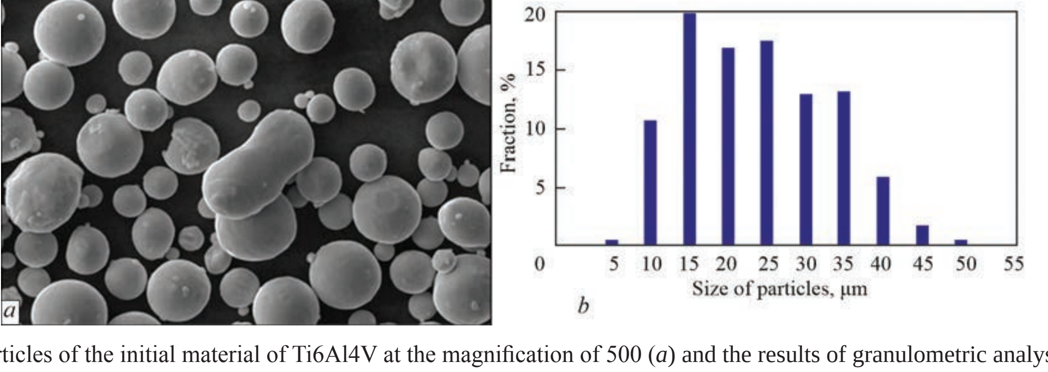

Figure 2. Particles of the initial material of Ti6Al4V at the magnification of 500 (*a*) and the results of granulometric analysis (*b*)

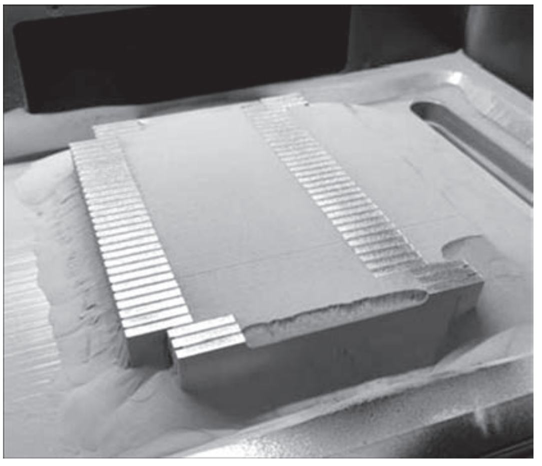

Figure 3. General view of manufactured experimental specimens

MATERIAL AND RESEARCH PROCEDURE

The studies were conducted on the specimens, manufactured of powder material using SLM technology. The printing of the specimens was conducted in the 3D printer Alfa-280 of the "ALT Ukraina" LLC [5, 6]. The material used in this study was titanium Ti6Al4V alloy with the size of particles from 5 to 40 μm. The chemical composition of Ti6Al4V powder in wt.% is the following: 6.21 Al; 4.03 V; 0.04 Fe; 0.1 C; 0.02 N; Ti is the base.

The initial material was investigated by means of the scanning electron microscope REM-106 (Figure 2, *a*) for determination of shape and sizes of particles. Figure 2, *b* shows the results of the analysis.

The experimental specimens for tensile tests were manufactured according to GOST 1497 — proportional flat specimens with the heads of type I of 3 mm thickness (Figure 3). The heat treatment was carried out at a temperature of 800 °C with an exposure of 1–5 h with a step of 1 h, the scheme of heat treatment is presented in Figure 4. Mechanical treatment of the specimens until reaching net sizes was performed using the lathe HAAS YT4.

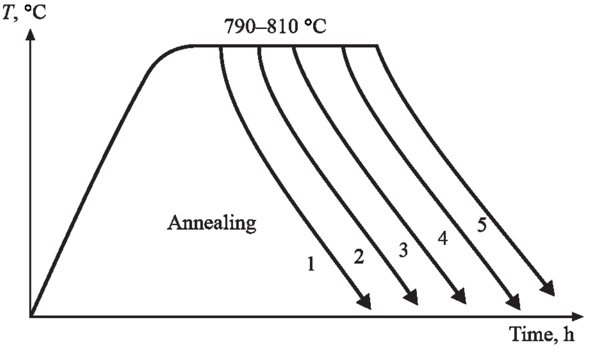

Figure 4. Scheme of heat treatment of experimental specimens

The heat treatment was carried out in the ShMP-27 shaft type furnace with the use of a protective environment (argon). The mechanical properties were determined during the tensile test by a standard procedure on the PHYWE machine. Metallographic sections were made according to standard procedures using diamond pastes. Microstructure examinations were performed with the use of Axiovert 200M optical microscope.

The statistical analysis was performed using a standard Excel package of data analysis.

EXPERIMENTAL RESULTS

According to the results of the analysis (Table 1), it was found that the values of mechanical properties after annealing are changed as compared to initial state after manufacturing. The comparative analysis of the values of ultimate strength of all experimental specimens after annealing at a temperature of 800 °C and with the range of 1–5 h exposures with a step of 1 h (specimens Nos 1–5) allows establishing a stable reduction in values by (–20.55– –23.03 %) with a slight discrepancy of ~2.5 % as compared to the initial state (specimen No. 6). Relative elongation after anneal-

Table 1. Mechanical properties of experimental specimens manufactured of titanium Ti6Al4V alloy using selective laser melting technology after heat treatment and in the initial state

Number	State	Ultimate strength, MPa	Δσ, %	Relative elongation, %	Δδ, %	Relative reduction in area, %	Δψ, %
1	Annealing at 800 °C, 1 h exposure	1003.73	–21.64	23.16	34.71	10.32	2.48
2	Annealing at 800 °C, 2 h exposure	989.987	–22.71	23.42	35.43	9.16	–9.03
3	Annealing at 800 °C, 3 h exposure	1017.65	–20.55	22.18	31.83	5.44	–45.97
4	Annealing at 800 °C, 4 h exposure	1008.47	-21.27	23.47	35.57	3.77	–62.56
5	Annealing at 800 °C, 5 h exposure	985.937	–23.03	22.02	31.33	6.4	–36.44
6	Initial state after manufacturing	1281	–	15.12	–	10.07	–

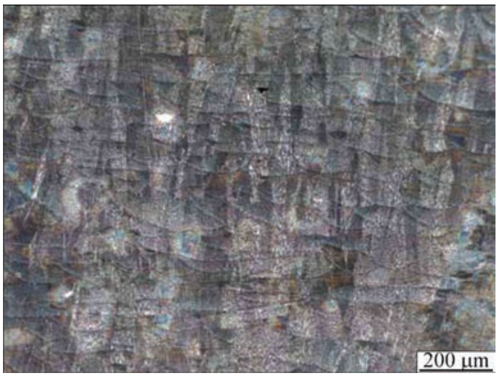

Figure 5. Structure of experimental specimen No. 6 (initial state after manufacturing)

ing undergoes stable changes (31.33–35.57 %) with a slight discrepancy of about 4 % as compared to the initial state. When analyzing changes in relative reduction in area, it was found, that annealing at 800 °C at 1 h exposure does not cause significant changes and with an increase in exposure time to 2, 3 and 4 h, a decrease in this characteristic by 9.03; 45.97; 62.56 % as compared to the initial state is observed. After 5 h exposure, the values of relative reduction in area undergo an increment of this characteristic as compared to the values after 4 h exposure by ~26.12 %, which may indicate changes in the microstructure of Ti6Al4V alloy.

As a result of the analysis of the values of mechanical properties, it was found that ultimate strength after heat treatment is reduced on average by ~21.84 % as compared to the initial state after manufacturing and relative elongation is increased by ~33.7 %.

The studies of experimental specimens in a polished state have shown that they all have a density of about 99.97 %, in most cases among defects there are separate globular pores with a diameter of 3–7 μm. During the microstructure examination, it was found that the experimental specimen No. 6 in the state after manufacturing has a scaly structure typical for the 3D process, which is formed as a result of solidification of individual melt pools. The microstructure was formed by α- and β-phases with columnar elongated grains, which grow crossing several layers (Figure 5).

According to the results of the microstructure examinations (Figures 6–11) of the heat-treated experimental specimens, it was found that the titanium alloy has α+β-structure. After exposure during 1 h (experimental specimen No. 1), a number of α-phase was 34.06 % (Figure 6), the ratio of the sides (width, length) of the α-phase lamella corresponded to ~ 0.5 (Figure 11).

While studying the ratio of the sides of the α-phase of the experimental specimens Nos 2–4, it was found that the α-phase has approximately the same geometric parameters and their ratio is in the range of 0.48–0.50, and its amount was 29.7, 31.11 and 30.87 % respectively (Figures 7–9, 11). The experimental specimen No. 5 has a structure α+β, while studying the α-phase, the ratio of the sides is 0.46, the percentage of which

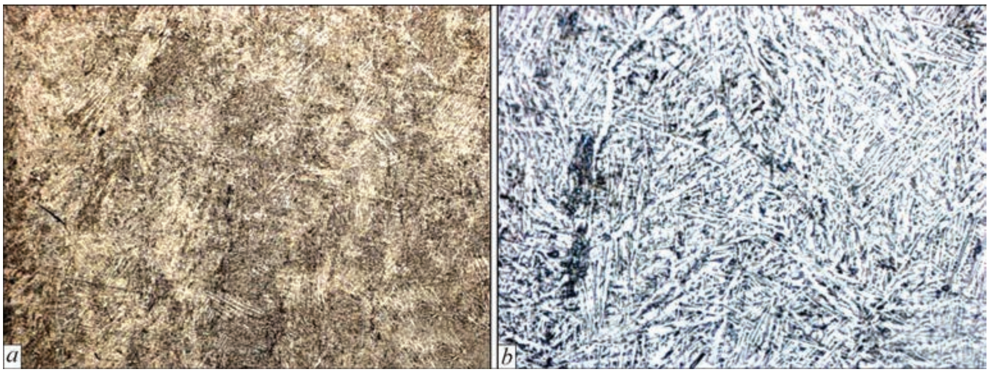

Figure 6. Structure of experimental specimen No. 1 after annealing at 800 °C with 1 h exposure: *a* — ×100; *b* — ×800

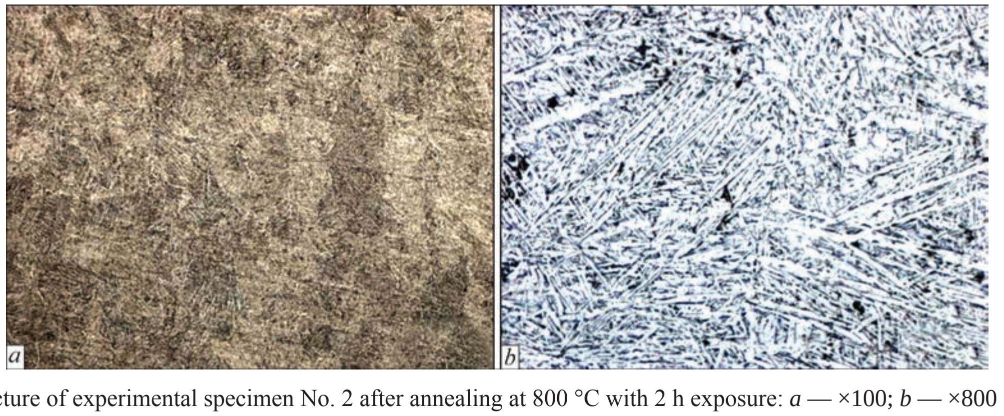

Figure 7. Structure of experimental specimen No. 2 after annealing at 800 °C with 2 h exposure: *a* — ×100; *b* — ×800

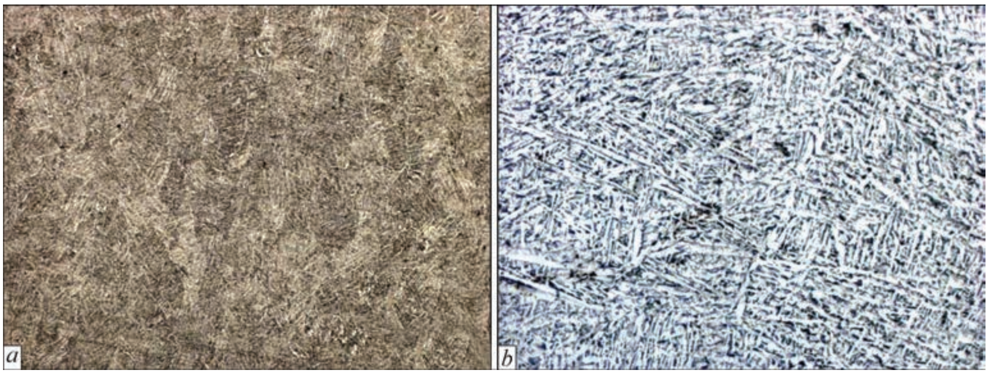

Figure 8. Structure of experimental specimen No. 3 after annealing at 800 °C with 3 h exposure: *a* — ×100; *b* — ×800

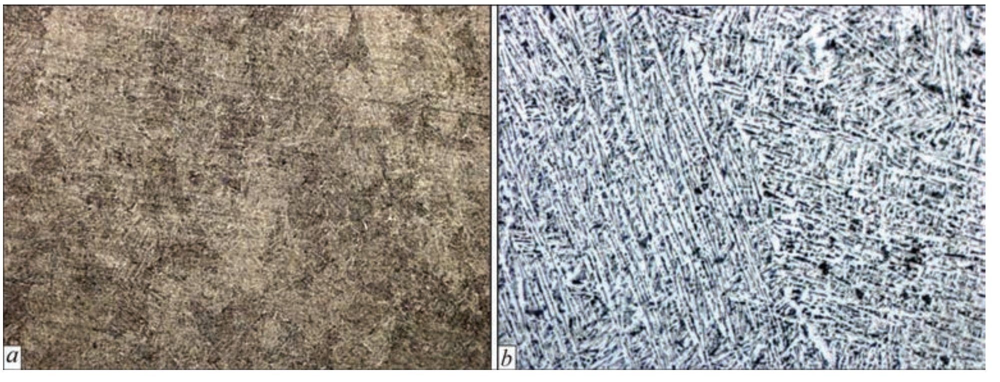

Figure 9. Structure of experimental specimen No. 4 after annealing at 800 °C with 4 h exposure: *a* — ×100; *b* — ×800

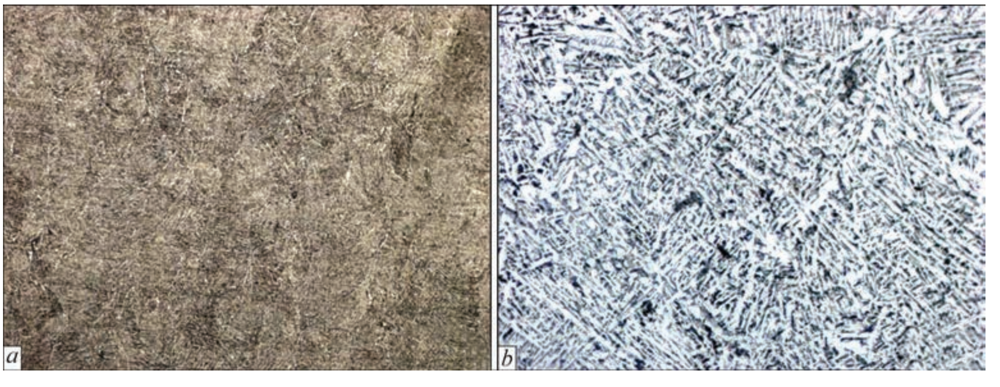

Figure 10. Structure of experimental specimen No. 5 after annealing at 800 °C with 5 h exposure: *a* — ×100; *b* — ×800

was 30.38 % (Figures 10, 11). According to the results of the microstructure examinations, the microstructure represents an α+β-structure with a slight change in the thickness of α-phase lamellas. According to the quantitative evaluation, it was found that the share of α-phase in all experimental specimens amounts to 29.7–34 %. It was found, that heat treatment with slow cooling, which is realized during cooling with a furnace, usually leads to the formation of α+β-lamellar microstructure and a small amount of equiaxial α-phase.

The results of the analysis of a paired correlation of the values of mechanical properties and parameters of the microstructure are presented in Table 2.

This criterion is used to measure the degree of linear dependence between two variables. The value of the coefficient of a paired correlation can vary from –1 to 1. At negative values of coefficient, the influence is negative and at positive values it is positive. At

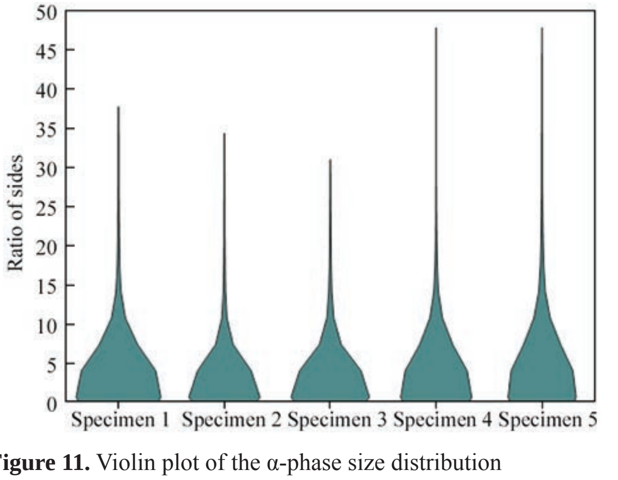

Figure 11. Violin plot of the α-phase size distribution

Table 2. Coefficients of paired correlation of mechanical properties and parameters of the structure after heat treatment of different duration

Parameter	Duration of annealing, h	Relative reduction in area, %	Ultimate strength, MPa	Relative elongation, %	Fraction of α-phase, %	Shape factor of α-phase
Duration of annealing, h	1	–	–	–	–	–
Relative reduction in area, %	–0.77806	1	–	–	–	–
Ultimate strength, MPa	–0.20611	–0.3955	1	–	–	–
Relative elongation, %	–0.50588	0.240275	0.014956	1	–	–
Fraction of α-phase, %	–0.58448	0.441543	0.372066	0.121644	1	–
Shape factor of α-phase	–0.53033	-0.09419	0.88632	0.437112	0.536141	1

values in the range of 1.0–0.5 (–1.0– –0.5), the ratio is considered high, at values of the coefficient in the range of 0.5–0.3 (–0.5– –0.3), the ratio is average, at 0.3–0.1 (–0.3– –0.1), the ratio is low, at lower values, the ratio is absent.

It was found that the ratio of the sides of α-phase precipitations has a high correlation with the values of ultimate strength, and the amount of α-phase is most correlating with the values of relative reduction in area at static tension.

CONCLUSIONS

1. As a result of the analysis of mechanical properties values it was found that ultimate strength after heat treatment of Ti6Al4V alloy, manufactured using the technology of selective laser melting, undergoes a decrease as compared to the initial state after manufacturing using the technology of selective laser melting by ~21.84 % and relative elongation was increased by ~33.7 %.

2. It was established that the heat treatment with slow cooling with the furnace of Ti6Al4V alloy, manufactured using the technology of selective laser melting, leads to the formation of α+β-lamellar microstructure and a small amount of equilibrium α-phase.

3. According to the results of the microstructure examination, it was found that after heat treatment, a change in the thickness of α-phase lamellas occurs, according to the quantitative evaluation it was found that the α-phase after 1–5 h exposure at 800 °C amounts to 29.7–34 %.

4. According to the analysis of results of the values of mechanical properties and parameters of the microstructure, a number and sizes of α-phase lamellas more significantly affects the relative reduction in area than other mechanical properties, shape factor has a close connection with the values of ultimate strength.

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ORCID

S.V. Adzhamskyi: 0000-0002-6095-8646,
G.A. Kononenko: 0000-0001-7446-4105,
V.R. Podolskyi: 0000-0002-0288-0641

CONFLICT OF INTEREST

The Authors declare no conflict of interest

CORRESPONDING AUTHOR

V.R. Podolskyi
E.O. Paton Electric Welding Institute of the NASU
11 Kazymyr Malevych Str., 03150, Kyiv, Ukraine.
E-mail: rostislavpodolskij@gmail.com

SUGGESTED CITATION

S.V. Adzhamskyi, G.A. Kononenko, V.R. Podolskyi (2022) Influence of heat treatment of specimens from Ti6Al4V manufactured by the technology of selective laser melting on structure and mechanical properties. *The Paton Welding J.*, **9**, 21–26.

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Received: 30.06.2022
Accepted: 11.11.2022