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Nanoindentation tests titanium alloy Ti-6Al-4V after interference laser treatment

Badania nanoindentacji stopu tytanu Ti-6Al-4V po interferencyjnej obróbce laserowej

MAGDALENA JAŻDŻEWSKA* ORCID: 0000-0002-3666-1551 SCOPUS AUTHOR ID: 56503890400
DOMINIKA B. TROCHOWSKA
MICHAŁ BARTMAŃSKI ORCID: 0000-0003-0185-6460 SCOPUS AUTHOR ID: 57194511871 RESEARCHERID: M-9589-2018

*Gdansk University of Technology,
Department of Material Engineering and Welding,
11/12 Gabriela Narutowicza Street, 80-233 Gdańsk
e-mail: magdalena.jazdzewska@pg.edu.pl

The study aimed was to assess selected mechanical properties of surface layers obtained after interference laser treatment – DLIL of titanium alloy Ti-6Al-4V used for implants. The samples were melted in still air at room temperature, for this purpose Nd:YAG laser was used, the number of laser shots was variable. Next, nanoindentation tests were performed, based on which Young's modulus and nanohardness in the obtained surface layers were determined. An increase in nanohardness is observed in the surface layers after laser melting. An increase in the modulus of elasticity and higher hardness was observed for both the first and second areas on the sample surface. In the case of a sample for which three laser beam shots were used, hardness and Young's modulus almost doubled. On the basis of the obtained results, it was found that the modified Ti-6Al-4V alloy can be used as an implant material, and the proposed method of modifying its surface may extend the life of such implants and improve their frictional properties.

Keywords: *Ti-6Al-4V alloy, interference laser treatment, nanoindentation*

Celem pracy była ocena wybranych właściwości mechanicznych warstw powierzchniowych otrzymanych po laserowej obróbce interferencyjnej – DLIL stopu tytanu Ti-6Al-4V stosowanego na implanty. Próbki nadtopiono w spokojnym powietrzu w temperaturze pokojowej, w tym celu wykorzystano laser Nd:YAG, ilość strzałów lasera była zmienna. Następnie wykonano badania nanoindentacji, na podstawie których określono moduł Younga oraz nanotwardość w otrzymanych warstwach powierzchniowych. Obserwowano wzrost nanotwardości warstw powierzchniowych po obróbce laserowej. Zaobserwowano wzrost modułu sprężystości i większą twardość zarówno dla pierwszego, jak i drugiego obszaru na powierzchni próbki. W przypadku próbki, dla której zastosowano trzy strzały wiązki laserowej, twardość i moduł Younga wzrosły prawie dwukrotnie. Na podstawie uzyskanych wyników stwierdzono, że zmodyfikowany stop Ti-6Al-4V może być stosowany jako materiał implantologiczny, a proponowana metoda modyfikacji jego powierzchni może wpływać na wydłużenie żywotności takich implantów oraz poprawiać ich właściwości cierne.

Słowa kluczowe: stop Ti-6Al-4V, interferencyjna obróbka laserowa, nanoindentacja

1. INTRODUCTION

In last time, titanium and its alloys have enjoyed increasing interest, especially in the field of biomaterial engineering. This biomaterial is used, among others in implantology for knee or hip joint prothesis, but also for facial plates. This is due to its fatigue strength, highest biotolerance and biocompatybility, and the lowest Young's modulus among currently used metallic biomaterials.

Nowadays, the most commonly used titanium alloy is Ti-6Al-4V, which, despite adequate high strenght, absences satisfactory biocompatibility. This is the result of the presence of elements such as aluminum and vanadium. Aluminum can cause Alzheimer's disease after being in the human body for a long time, while vanadium can cause ailment such as perpheral neuropathy or osteomalacia. For Ti-6Al-4V alloy to be used safety in implantology, it must be surface treated. It will improve the mechanical properties and increase the hardness of the material [1–3].

Laser radiation is increasingly used for surface treatment of materials, because high energy density can be used and the amount of energy delivered directly to the surface area can be minimized. Thanks to that you can be avoided among other distortion of worked element. The use of a laser enables processing of irregularly shaped and large dimensioned objects. The laser power can be adjusted during the process, and the treatment does not need to be carried out in a vacuum or other protective atmosphere. The disadvantage of this process is the high cost of the equipment needed [4].

Laser ablation is used in micro- and nanotreatment of material's surfaces, including creating structures 1D, 2D and 3D. One of the newest technologies using the phenomenon of interference of laser beams to produce periodic micro- and nanometric matrices on the surface of hard materials is direct laser interference lithography (DLIL). It allows complete control of the obtained matrix dimensions (width and height of the recesses as well as periodicity of distribution) on the modified surfaces by constant control of the laser beam parameters [5, 6].

DLIL is based on selective laser ablation, melting or recrystallization of the surface layer in interference maxima. Therefore fringes or holes are obtained in micro- or nanoscale. The use of a pulsed laser with high power density (from MW/cm^2 to GW/cm^2), allows the radiation of almost any type of solid surface. However, small angles of incidence of the laser beams on the sample (θ = 1–5°) and the beams with a diameter of several millimeters, allow the impact on an area of many mm^3. DLIL is particularly used for modification titanium alloys Ti-6Al-4V and Ti-13Nb-13Zr and amorphous carbon (DLC) [5, 6].

2. EXPERIMENTAL

Two-phase (*α+β*) Ti-6Al-4V titanium alloy with the chemical composition as shown in Table 1 was tested. Usually used in an annealed condition at temperatures up to 399°C. The alloy has a differential chemical composition, because during the production of the amount of component elements are subjected to modifications. In addition to aluminum in the range 5,5–6,75% of content and vanadium 3,5–4,5%, there are also small amounts of other elements (oxygen to 0,2% and nitrogen to 0,05%). An increase in the oxygen and nitrogen content increases the strength of the material, and a smaller proportion of theses elements gives adequate plasticity and resistance to cracking [7].

Samples were cut from the company's titanium sheets „Timet". The sheet was delivered in an annealed condition at temperature 750°C. The samples had a cuboid shape with dimensions 8 mm × 12 mm × 18 mm. Preparation of the samples consisted of surface grinding up to a gradation of 2000, cleaning the surface of impurities and polishing with diamond paste with grains size 2 μm.

The titanium alloy Ti-6Al-4V tested has high strength even to 315°C and a melting point in the range 1610–1660°C. It also has a high ratio of strength to a material density and very good corrosion resistance. Also interesting is the yield strength, which is 900 MPa for microcrystalline titanium and 1040 MPa for nanocrystalline titanium. For medical applications, it is most often used at the nanometer scale because the surface of nanocrystalline sinters facilitates contact with osteoblasts and faster mineralization [8]. The selected mechanical properties of titanium alloy Ti-6Al-4V are shown in Table 2.

The prepared sample has been surface modified at the Institute of Optoelectronics of the Military University of Technology in Warsaw. During processing, a dual-channel Nd:YAG laser system with Q-modulation was used. To obtain fast shaping of periodic structures on a surface of several cm^2, laser beams with an inch front set in a quasiflat. This arrangement of the beams resulted in a Mach-Zehnder interferometer. The supplied power in a single channel pulse was 171 mJ, the pulse time was 8 ns and the frequency 10 Hz. Two slightly different treatments were performed. They differed in the number of longitudinal modes. Two laser shots (N=2 – DLIL 1) were used for the first treatment and three shots (N=3 – DLIL 2) for the second [5, 9]. The parameters of laser treatments are shown in Table 3.

Table 1. Chemical composition of Ti-6Al-4V alloy
Tabela. 1. Skład chemiczny próbki stopu Ti-6Al-4V

Element	V	C	Fe	N	Al	O	H	B	Y	Ti
ISO 5832/3, % mass	3.5–4.5	<0.08	<0.30	<0.05	5.50–6.75	<0.20	<0.0015	-	-	rest
Atest, % mass	4.05	0.01	0.16	0.005	6.40	0.185	0.0035	max. 0.001	max. 0.001	rest

Table 2. Selected mechanical properties of titanium alloy Ti-6Al-4V
Tabela. 2. Wybrane właściwości mechaniczne stopu tytanu Ti-6Al-4V

Rolling direction	Re, MPa	Rm, MPa	A5, %
Longitudinal	1050	1072	9.53
Transverse	1002	1023	9.49
Acc. to ISO 5832/3	min. 830	900–1160	min. 10

Table 3. Parameters of laser treatment
Tabela. 3. Parametry obróbki laserowej

Test parameter	Singel channel pulse, mJ	Pulse time, ns	Frequency, Hz	Number of laser shot
DLIL 1	171	8	10	2
DLIL 2	171	8	10	3

For nanoindentation research prepared nanoindenter British company MicroMaterials (NanoTest) Vantage. The applied nanoindentation study allowed the determination of such mechanical properties as modulus of elasticity and nanohardness. Before the experiment, there were performed a calibration lasting 20 s and a temperature drift for 60 s. The first test of hardness and elasticity modulus was performed for the base material of titanium alloy Ti-6Al-4V, and then for the first and second area on the surface that was subjected to Direct Laser Interference Lithography. The test parameters used are shown in Table 4 [10].

Table 4. Parameters of nanoindentation test
Tabela. 4. Parametry badania nanoindentacji

Test parameter	Value
Quantity of indentations	50
Maximum force value, mN	10
Rise time, s	20
Hold time with maximum force, s	10
Unloading time, s	15
Distance between indentations, μm	20

3. RESULTS AND DISCUSSION

The results of elasticity modulus and nanohardness measurements of Ti-6Al-4V and after modification samples are shown in Table 5. Figures 1–3 are presented single hysteresis load-deformation graphs for the base material and the remelted samples: DLIL 1 and DLIL 2. Each of the curves consists of three sections: increasing the force to the maximum value, holding with maximum force, and offloading. The obtained values of the elasticity modulus and nanohardness for areas after DLIL increased in comparison with the values of these parameters for the base material. It was also observed that when using an additional laser shot for the DLIL 2 sample, Young's modulus and nanohardness were twice the values compared to the DLIL 1 sample. Therefore, it can be concluded that the choice of the number of laser shots during direct laser interference lithography has a big impact on the tested mechanical properties, i.e. Young's modulus and nanohardness.

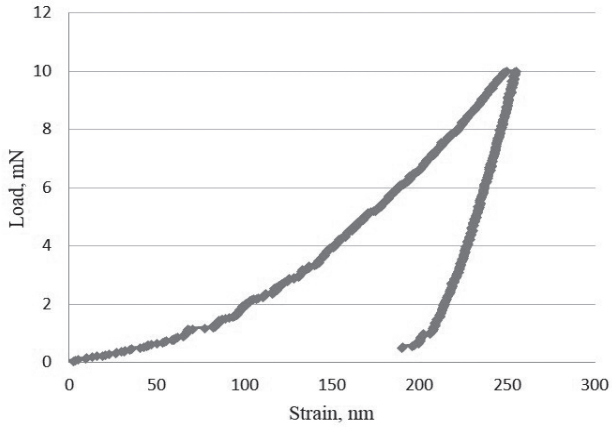

Fig. 1. Sample hysteresis curve load–strain for base material
Rys. 1. Przykładowa krzywa histerezy obciążenie–odkształcenie dla materiału rodzimego

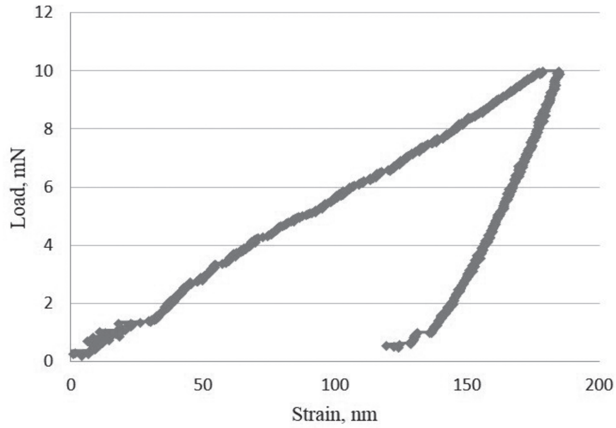

Fig. 2. Sample hysteresis curve load–strain for DLIL 1
Rys. 2. Przykładowa krzywa histerezy obciążenie–odkształcenie dla próbki DLIL 1

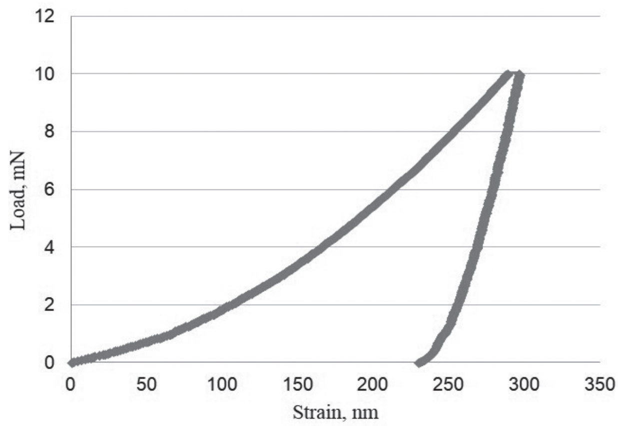

Fig. 3. Sample hysteresis curve load–strain for DLIL 2
Rys. 3. Przykładowa krzywa histerezy obciążenie–odkształcenie dla próbki DLIL 2

Table 5. Values of the elasticity modulus and nanohardness of the tested base material and the modified areas tested on the surface of the Ti-6Al-4V alloy sample
Tabela. 5. Wartości modułu sprężystości i nanotwardości badanego materiału rodzimego i zmodyfikowanych obszarów na powierzchni próbki Ti-6Al-4V

Test parameter	BM	DLIL 1 (N=2, power 2×171 mJ)	DLIL 2 (N=3, power 2×171 mJ)
Reduced modulus of elasticity, GPa	(131.0–5.6)	(170.71–19.23)	(240.22–49.01)
Modulus of elasticity, GPa	(137.12–4.98)	(186.43–25.00)	(286.04–81.80)
Nanohardness, GPa	(3.95–0.35)	(6.86–1.39)	(13.31–5.84)

4. CONCLUSIONS

The analysis of the results obtained during the nanoindentation study showed that the values of Young's modulus and nanohardness for areas after direct laser interference lithography increased compared to the base material. It also turned out that the nanohardness and modulus of elasticity for the DLIL 2 sample are twice as high as compared to the DLIL 1 sample. The increase in the value of the tested mechanical properties was caused by the use of an additional laser shot for the DLIL 2 sample. Therefore, it is concluded that the choice of the number of laser shots has an impact on hardness and Young's modulus.

The divergence of the obtained values of the tested parameters is a confirmation of a very well-developed periodic structure that was created as a result of the laser processing. This is also the reason for high measurement uncertainties, designatedas the standard deviation.

Analysis of the results of the research described in this paper entitled for the following conclusion that the modification applied leads to the possible use of Ti-6Al-4V alloy as an implantology material. The use of the DLIL process has resulted in higher values of nanohardness and modulus of elasticity compared to the base material. Due to increased resistance to wear, it is also possible to extend the life of biomaterials made of Ti-6Al-4V alloy.

REFERENCES

[1] Ashby M., Shercliff H., Cebon D.: Materials engineering, science, processing and design. Elsevier Ltd., Cambridge (2010) 2.

[2] Głowacka M., Łabanowski J.: Inżynieria powierzchni. Wybrane zagadnienia. Wydawnictwo Państwowej Wyższej Szkoły Zawodowej w Elblągu, Elbląg (2014).

[3] Chorzelska M., Jażdżewska M.: Utlenianie anodowe biostopu Ti6Al4V w aspekcie jego zastosowań w implantacji. Inżynier i Fizyk Medyczny 5 (7) (2018) 317–319.

[4] Biernat J.F.: Materiałoznawstwo. Wydawnictwo Politechniki Gdańskiej, Gdańsk (2014).

[5] Czyż K., Strzelec M., Marczak J., Rycyk A., Sarzyński A., Major R., Patron Z., Jach K., Świerczyński R., Wachulak P., Garbacz H.: Bezpośrednia laserowa litografia interferencyjna w periodycznej obróbce biomateriałów. Biuletyn WAT 4 (LXV) (2016) 31–53.

[6] Czyż K., Major R., Trembacka-Wójciga K., Rycyk A., Sarzyński A., Strzelec M., Marczak J.: Periodyczne strukturowanie powierzchni wybranych materiałów biozgodnych. Przegląd Elektrotechniczny 5 (91) (2015) 49–52.

[7] Welsch G., Rodney B., Collings E.W.: Materials properties handbook. Titanium alloys. ASM International (1993) 8.

[8] Kurzydłowski K., Lewandowska M.: Nanomateriały inżynierskie konstrukcyjne i inżynierskie. Wydawnictwo Naukowe PWN, Warszawa (2010) 9.

[9] Majkowska B., Jażdżewska M., Zieliński A., Antoszkiewidz M., Marczak J., Sarzyński A., Strzelec M., Rycyk A., Czyż K.: Influence of laser interference micromachining of Ti13Nb13Zr bio-alloy on adhesion of hydroxyapatite coatings. Symposium R.: Bioceramics for Bone and Joint Repair. Warszawa (2014).

[10] Oliver W.C., Pharr G.M.: Measurement of hardness and elastic modulus by instrument indentation. Advances in under standing and refinements to methodology. Journal of Materials Research 19 (2004) 3–20.