Influence of the protective atmosphere on the structure and properties parts from titanium alloy Ti-6Al-4V produced by direct laser deposition

To cite this article: M O Gushchina et al 2018 J. Phys.: Conf. Ser. 1109 012060

View the article online for updates and enhancements.
Influence of the protective atmosphere on the structure and properties parts from titanium alloy Ti-6Al-4V produced by direct laser deposition

M O Gushchina\textsuperscript{1,2,*}, O G Klimova-Korsmik \textsuperscript{1,2}, A M Vildanov, S A Shalnova \textsuperscript{2}, A S Tataru\textsuperscript{3}, E A Norman\textsuperscript{1,2}

\textsuperscript{1} St. Petersburg State Marine Technical University, St. Petersburg, Russia
\textsuperscript{2} St. Petersburg Polytechnic University, St. Petersburg, Russia
\textsuperscript{3} National University of Science & Technology (MISIS), Moscow, Russia

*Corresponding author e-mail: skmar.spb@gmail.com

Abstract. This work is devoted to the microstructure, tensile deformation and microhardness behaviour of Ti-6Al-4V alloy fabricated using direct laser deposition additive manufacturing with using different oxygen concentration in protected chamber. The results show that the microstructure consists of coarse columnar prior-\textgamma\textsuperscript{-}phase grains and \textalpha\textsuperscript{-}laths, with decreasing of oxygen concentration a \textbeta\textsuperscript{-}phase amount is reduce. Ti-6Al-4V alloy has a basket-weave microstructure in the atmosphere with concentration of oxygen up to 0.25\%, also it has higher concentration martensitic structure. Using local protection it is impossible to achieve the necessary mechanical characteristics. The required properties for Ti-6Al-4V are achieved with an oxygen content in the chamber in the range of [0.02-0.25]\%, further reduction of the content is meaningless, since the mechanical properties vary slightly and higher 0.5 mechanical properties of samples is decreased.

1. Introduction

Titanium based alloys have properties due to which they find wide application in the aviation, shipbuilding, petrochemical industry. Titanium alloys combine high specific strength, high corrosion and erosion resistance, crack resistance and resistance to fatigue loads, practically do not oxidize up to temperatures of 400\degree C \textsuperscript{1}. Despite all these advantages due to the low thermal conductivity of titanium alloys, it is difficult to machining. This is important flaw of this alloy. This is especially important in the production of large-sized monolithic parts with complex shapes, which are used in aircraft construction and shipbuilding \textsuperscript{[2]}. Machining of these alloys leads to increased manufacturing complexity. For example, the complexity of processing parts and aggregates of aircraft is 25-35\% of the total labor intensity of production. To improve productivity and economic efficiency of enterprises, it is necessary to use new technologies that reduce the consumption of raw materials and labor. Most perspective manufacturing process in this fields is additive technologies \textsuperscript{[3-6]}. This class of technologies is based on principle of adding material.

High Speed Direct Laser Deposition Technology is perspective technology for producing large-scale parts. Equipment for HS DLD is universal and based on modularity principle \textsuperscript{[7,8]}. Without additional tools and equipment, it is possible to receive part of any designs and complexity at the
request of the customer. This technology allows reducing the raw materials consumption that spent on parts production, as well as reduce the amount of manufacturing waste [9,10].

Attractiveness of titanium alloys is due to high strength-to-weight ratio, low capacity and high activity in interaction with the atmosphere gases in a state heated to 600-800°C [11]. The last fact is important for DLD technology at the producing parts from titanium alloys. It requires sealed chamber that provides a sufficient level of purity argon. The design of the chamber requires understanding of the effect of oxygen on the structure and mechanical properties of the samples. In order to establish the dependence of the quality of manufactured products on the quality of the protective atmosphere, a number of experiments were performed with varying the percentage of oxygen in the protective chamber.

2. Experimental procedure
Experimental research was carried out on laboratory setup for DLD based on fiber laser LS 5(5kW) produced in Institute of Laser and Welding Technology (ILWT) (figure 1a). The laboratory setup consists from powder feeder, welding laser head, coax nozzle, chiller. As a manipulator, the 6-axis robotic system manufactured by Fanuc corp is used[8].

Mechanical tests were carried out on samples obtained with different oxygen concentrations in the chamber. Laser power level is 1350W, process speed is 25 mm/s, coax gas consumption is 18 l/min. Variation of oxygen concentration shown in table 1.

| Sample number | A1 | A2 | A3 | A4 | A5 | A6 |
|---------------|----|----|----|----|----|----|
| Oxygen concentration, % | Local protect | 1 | 0,5 | 0,25 | 0,1 | 0,02 |

Protection gas is high purity argon (99,98%). Fractional composition is 40-200 microns, the shape of the particles is spherical. The powder was made by the method of gas atomization. Chemical composition is shown in table 2

| Ti, % | Al, % | V, % | Fe, % | Zr, % | O, % | H, % | N, % |
|-------|-------|------|-------|-------|------|------|------|
| Bal.  | 5,3-6,8 | 3,5-5,3 | to 0,6 | to 0,3 | to 0,2 | to 0,015 | to 0,05 |

Metallographic studies were carried out on microscope DMI 5000 (Leica) with Tixomet software. Researches of chemical composition and chemical elements distribution were made on scanning electron microscope PhenomProX, Mira Tescan and Lira Tescan microscopes using console Oxford.
INCA Wave 500. For determining of mechanical properties samples were tested on uniaxial tension, using universal testing machine Zwick/Roell Z250 Allround.

Researches of the phase composition and structural parameters of the samples were carried out on a XRD-6000 diffractometer on CuKα radiation. The analysis of the phase composition was carried out using PDF 4+ databases, as well as the full-profile analysis program POWDER CELL 2.4.

3. Results
The Ti–6Al–4V alloy is high strength, two-phase (α+β) titanium alloys. Aluminum addition stabilizes and strengthen α phase, increases α+β↔β transformation temperature and reduces alloy density. Vanadium – β-stabilizer – reduces α+β↔β transformation temperature and facilitates hot working (higher volume fraction of β-phase) [12,13]. Oxygen and nitrogen have wide solubility region with α-titanium and decreasing polymorphic transformation temperature.

Deposited samples produced with different oxygen concentration in protected chamber have heat colors on the surface - TiO₂ formed on sample surface during reaction between oxygen and titanium (figure 2). Color is depends on oxide layer thickness. According with requires for welding process of titanium alloys brown, blue, purple and green colors are unacceptable.

![Figure 2. Samples produced with using different concentration of oxygen in the chamber local protection](image)

3.1. Microstructure characterization
In the cooling process Ti-6Al-4V alloy from β phase range α-phase growing begin below β-transformation temperature (980°C). The α-phase is formed as lamellae which have crystallographic relations with β-phase [14]. Although β-grains during polymorphic transformation β → α are divide to several finest grains, according with orientation principle each α-grain have common in direction of orientation with adjacent α-grains. As the result so called intergranular structure especial grain orientation are formed. Rapid solidification leads to martensitic decomposition of β phase and formation Widmanstatten structure α-phase.

During direct laser deposition using local argon protecting martensitic structure with acicular α’-phase is observed (thin lamellae oriented perpendicular to each other). But an advantageous balance of properties can be obtained by development of bimodal microstructure consisting of primary α-grains and fine lamellar α colonies within relatively small β-grains (10-20 µm in diameter) (figure 3).
Figure 3. Microstructure of Ti-6Al-4V produced by DLD with A1 (local protection) (a) A2 (1%O₂) (b) A3 (0.5%O₂) (c) A4 (0.25%O₂) (d) A5 (0.1%O₂) (e) A6 (0.02%O₂) (f) μ

With increasing of oxygen content, the structure of the lamellas, \( \alpha + \beta \) phase, a martensite \( \alpha \) phase is observed with a decrease in the oxygen content and local protection. Structural differences can be also associated with thermal convection heating from a heated gas. It occurs when there is a long exposure to the laser radiation during deposition process, which provides additional heat input, and the sample cooling is slower than local gas protection.

Decreasing of oxygen concentration in protection chamber leads to growing of lamellae wide and length, content of \( \beta \)-phase is increasing too. This may be due to the fact that oxygen and nitrogen is \( \alpha \)-stabilized elements and they increase temperature of \( \alpha \rightarrow \beta \) transformation.
3.2. Mechanical properties

3.2.1. Tensile properties.

In table 3 tensile stress-strain result of the Ti-6Al-4V at a content of oxygen in chamber 1, 0.5, 0.25, 0.1, 0.02% and local protection is shown. Samples was tested with six levels of oxygen concentration.

| Sample | Yield strength, (MPa) | Tensile strength, (MPa) | Elongation, (%) | Oxygen concentration, (%) |
|--------|-----------------------|-------------------------|-----------------|---------------------------|
| A1     | 934,4                 | 983,4                   | 1,6             | Local protect             |
| A2     | 872,7                 | 929,0                   | 3,3             |                           |
| A3     | 835,7                 | 916,7                   | 5,3             | 0,5                       |
| A4     | 811,3                 | 841,8                   | 7,2             | 0,25                      |
| A5     | 779,8                 | 819,9                   | 5,5             | 0,1                       |
| A6     | 813,0                 | 856,3                   | 6,0             | 0,02                      |

As mentioned above, growth of oxygen concentration until 0,25%O2 leads to change of mechanical characteristics. A tensile strength is decrease and ductility is increase with reduce gasses concentration in protect chamber (table 3). Using concentration below 0,25% value of elongation and strength reduction is observed. But the best mechanical properties by strength to ductility ratio have sample A4, which was produced with 0,25% oxygen concentration in chamber.

3.2.2. Fracture morphology

The fractographic investigation was conducted using scanning electron microscopy for determination a source of destruction. The results are shown on figure 4.

Figure 4. Fracture morphology of DLD Ti-6Al-4V sample (a) A1(local protection) (b) A2 (1%O2) (c) A3 (0,5%O2) (d) A4 (0,25%O2) (e) A5 (0,1%O2) (f) A6 (0,02%O2)
Fracture surface shows the ductile fracture in the deposition Ti-6Al-4V samples when under uniaxial tensile stress. Furthermore, the samples A2, A3, A5 had greater and deeper dimples in the fracture surfaces than the A4, A6 samples indicating that the sample with oxygen concentration 0,25% and 0,02% had better ductility than the sample with oxygen concentration 1%, 0,5%, 0,1% . Fracture of sample, which was produced under local protection, consists dark grey plate inclusions (supposedly inclusion is titanium hydride). According to mentioned above, a destruction of A1 sample (using local protection) occurs due to inclusions formation.

3.2.3. Microhardness

Table 4 presents the values of Vickers hardness is measured in A1-A6 samples (figure 5a). The microhardness of the produced zone, average value and reference value range is presented in table 4. Figure 5b shows a microhardness comparison of samples produced with different oxygen concentration in protect chamber.

Table 4. Microhardness (HV) of the test sample

| Point number | Microhardness, (HV0,5) |
|--------------|------------------------|
|              | A1 | A2 | A3 | A4 | A5 | A6 |
| 1            | 343| 362| 365| 366| 370| 395|
| 2            | 341| 362| 366| 364| 384| 394|
| 3            | 340| 362| 356| 359| 392| 407|
| 4            | 355| 365| 378| 361| 387| 396|
| 5            | 349| 364| 367| 370| 397| 408|
| 6            | 340| 363| 362| 376| 389| 429|
| 7            | 352| 362| 359| 362| 388| 409|
| 8            | 342| 365| 363| 369| 396| 411|
| 9            | 345| 362| 360| 359| 397| 411|
| 10           | 343| 364| 366| 358| 362| 401|
| Average value| 345| 363,1| 364,2| 364,4| 386,2| 406,1|
| Reference value range[14] | 257-373 |
It can be seen that hardness value is increase with decreasing of oxygen concentration. Used oxygen concentration below 0,25% lead to increase hardness to value above reference value range.

3.3. XRD analyses
When concentration of oxygen in chamber is increase β-phase content is decrease and quantity of α/α’ is increase. The greatest content of β-phase is observed with local protection. Probably it due to the difference of cooling cycles in comparison with the samples obtained in the chamber with argon protection (figure 6).
4. Conclusions
The relationship between the quality of manufactured parts from Ti-6Al-4V alloy and quality of the protective atmosphere during direct laser deposition was investigate using oxygen concentration control in protective chamber.

Growth of oxygen concentration until 0.25%O2 leads to decreasing of tensile strength and increasing of ductility. Using concentration below 0.25% a slight decrease in the relative elongation and strength is observed. Best mechanical properties by strength to ductility ratio sample A4 has, which was produced with 0.25% oxygen concentration in chamber. The best mechanical properties by strength to ductility ratio sample has, which produced with 0.25% oxygen concentration in chamber.

Fracture surface shows the ductile fracture in the deposition Ti-6Al-4V samples when under uniaxial tensile stress. Fracture of sample, which was produced under local protection, consists dark grey plate inclusions (supposedly inclusion is titanium hydride).

Hardness value is increases with decreasing of oxygen concentration. Used oxygen concentration below 0.25% leads to increase hardness to value above reference value range. With local protection, The greatest content of β-phase is observed under the local protection. Probably it due to the difference of cooling cycles in comparison with the samples obtained in the chamber with argon protection.

Using local protection it is impossible to achieve the necessary mechanical characteristics. The required properties for aTi-6Al-4V are achieved with an oxygen content in the chamber in the range of [0.02-0.25]%.

Acknowledgment
The work was carried out with financial support from the Government of the Russian Federation (Ministry of Defense) in the framework of realization complex project Contract No 03.G25.31.0240 on 28.04.2017.

References
[1] Jun Yu, Rombouts Marleen, Maes Gert, Motmans Filip 2012 Phys. Proc. 39 416-24
[2] Qiu C, Ravi G A, Dance C, Ranso A, Dilworth S, and Attallah M M 2015 Proc. CIRP 35 55
[3] Uhlmann Eckart, Kersting Robert, Kleina Tiago Borsoi, Fernando Cruz Marcio, Vicente Borille Anderson 2015 J. of All. and Comp. 629 351
[4] Keist J S, Palmer T A 2016 Mater. and Des. 106 482
[5] Ren Y M, Lin X, Fu X, Tan H, Chen J, Huang W D 2017 Acta Mater. 132 82
[6] Sklyar M O, Klimova-Kormik O G Cheverikin V V 2016 Sol. St. Phen. 265 535
[7] Turichin G A, Klimova O G, Zemlyakov E V, Babkin K D., Kolodyazhnnyy D Yu, Shamray F A, Travyanov A Y, Petrovskiy P V 2015 Phys. Proc. 78 397
[8] Turichin GA, Somonov V V, Babkin K D, Zemlyakov E V, Klimova O G 2016 High-Speed Direct Laser Deposition: Technology, Equipment and Materials Mater. Sci. and Eng. 125
[9] Shamsaei Nima.,, Yadollahia Aref, Bian Linkan, Thompson Scott M. 2015 Add. Manuf. 8 12-35
[10] DebRoy T, Wei H L, Zuback J S, Mukherjee T, Elmer J W, Milewski J O, Beese A M, Wilson-Heid A, De A, Zhang W 2018 Prog.in Mat. Sci. 92 112
[11] Donachie Matt. J, 1988 Titanium, A technical Guide, ed. Donachie Matt. J (Ohio: ASM International)
[12] Sieniawski J 2013 Titanium alloys - Advances in Properties Control, InTech, Ed: Jan Sieniawski, Waldemar Ziaja (London:InTech) chapter Microstructure and mechanical properties of high strength two-phase titanium alloys pp.69-80
[13] Wu X., Liang J., Mei J, Mitchell C, Goodwin P S, Voice W 2004 Mater. and Des. 25 137-144
[14] Donachie Matt. J, 2000 Titanium, A technical Guide 2nd ed, ed. Donachie Matt. J (Ohio: ASM International)