Forming a single layer of a composite powder based on the Ti-Nb system via selective laser melting (SLM)

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Abstract: Alloys based on the titanium-niobium system are widely used in implant production. It is conditional, first of all, on the low modulus of elasticity and bio-inert properties of an alloy. These alloys are especially important for tooth replacement and orthopedic surgery. At present alloys based on the titanium-niobium system are produced mainly using conventional metallurgical methods. The further subtractive manufacturing an end product results in a lot of wastes, increasing, therefore, its cost. The alternative of these processes is additive manufacturing. Selective laser melting is a technology, which makes it possible to synthesize products of metal powders and their blends. The point of this technology is laser melting a layer of a powdered material; then a sintered layer is coated with the next layer of powder etc. Complex products and working prototypes are made on the base of this technology. The authors of this paper address to the issue of applying selective laser melting in order to synthesize a binary alloy of a composite powder based on the titanium-niobium system. A set of 10x10 mm samples is made in various process conditions. The samples are made by an experimental selective laser synthesis machine «VARISKAF-100MB». The machine provides adjustment of the following process variables: laser emission power, scanning rate and pitch, temperature of powder pre-heating, thickness of the layer to be sprinkled, and diameter of laser spot focusing. All samples are made in the preliminary vacuumized shielding atmosphere of argon. The porosity and thickness of the sintered layer related to the laser emission power are shown at various scanning rates. It is revealed that scanning rate and laser emission power are adjustable process variables, having the greatest effect on forming the sintered layer.

1. Introduction

Titanium and its alloys are widely-used materials for manufacturing medical implants and surgical instruments. Tooth replacement and orthopedic surgery are the fields, where these alloys are used most frequently (Figure 1) due to their good mechanical properties, bio-compatibility and high corrosion resistance.



Figure 1. Tooth implant produced of titanium alloy [1].

The high modulus of elasticity (above 100 GPa) is a disadvantage of titanium and alloys on its base. Therefore, the main part of mechanical load is applied to the bones, and not to the implant, resulting in bone tissue resorption (Figure 2), implant weakening and reoperation [2]. At present, search and development of alloys with the low modulus of elasticity, having high strength and sufficient product plasticity though, are in focus of the scientists [3, 4].



Figure 2. Resorption (failure) of a bone around the implant [5].

Alloys of the titanium-niobium system are distinguished by the most appropriate combination of mechanical properties. Moreover, niobium and titanium are non-toxic elements and do not react with body tissues. Some alloys of the titanium-niobium system have the low modulus of elasticity.

The conventional metallurgical methods of manufacturing titanium alloys are imposed by the technical and principal restrictions [6]. Titanium and cobalt-chromium alloy implants are manufactured via casting with further milling or turning, using methods of powder metallurgy, hot stamping, and stamping with further milling.

Casting is also a method to produce implants. High-frequency casting machines, providing reliable quality of casts, are used for melting steel or cobalt-chromium alloys. As a rule, producing implants of cobalt-chromium alloys is not difficult in laboratory conditions. However, casting implants of titanium-based alloys is more difficult because titanium reacts with nitrogen and oxygen when heating in air, and with form lining in the molten state. The technology of casting is based on vacuum melting and casting, and unconventional high fireproof forms are required as well. Therefore, titanium implants with a good quality surface can be produced in industrial conditions only. Cast implants are subject to X-ray diffraction control, revealing up to 5-8 % of wastes [7].

Laminar implants can be milled only when developing new titanium structures. In mass production milling an implant of a metal sheet is non-value-added. Spiral, cylindrical implants can be produced via turning only.

Hot stamping is a principal procedure of metal processing, including titanium and cobalt-chromium alloys. However, it should be noted implants are small products, and their gas absorbability grows when heating, therefore, technological procedures are strictly standardized. Equipment for stamping is quite complex and expensive, so it can be used when mass-manufacturing critical parts.

The procedure based on stamping and milling is technologically simple, so mass-manufacturing of products is more profitable. Test-production of titanium implants has revealed that it is the most appropriate and acceptable variant. The production of implants comprises two phases. The first phase includes cutting implant half-products by a special stamp from a 3 mm thick titanium sheet. The second phase is aimed at simultaneous thinning the implant base on both sides up to 1.2 mm by a milling machine. As burrs are removed, the implant is prepared for electrochemical polishing [7, 8].

These days the methods of layer-by-layer laser synthesizing end products of various powder materials are being successfully adopted by the health care system - one of the most important fields of human activity. It is mainly because the manufacturing cycle of the implant is shorter than that of conventional processing methods (milling, turning etc.), it is possible to save expensive materials and manufacture products of a complex geometry and specified porosity as well [9, 10].

Selective laser melting is a procedure of manufacturing a 3D product via successive laser beam fusion of powder materials according to a specified program (Figure 3).



Figure 3. A layout of selective laser melting process [11].

Selective laser melting supports selecting and monitoring all basic parameters, which affect the process of product synthesizing such as emission power, scanning rate, and temperature of powder heating [11-13]. So it is possible to manufacture end products with specified mechanical characteristics [14].

The process includes two phases: first, an even thin layer of powder is spread all over the working area, then the laser is switched on; and the areas similar to the section of object to be produced are melted. A piston goes down approximately as deep as thick the layer is, then, the procedure is repeated, as soon as the top point of the model is approached.

Process parameters are adjustable at each phase of selective laser melting. To put the powder one can use a roller or a scraper. Melting is possible on the outer contour of the layer only or over the complete depth of the model.

Selective laser melting requires no supporting structure as a big amount of powder around the model keeps it from destruction, unless the end form is acquired and the mechanical article strength is achieved [15-17].

2. Methods and equipment of experimental research

A SLS machine «VARISKAF-100MB» is used for selective laser melting a composite powder containing 60 wt. % Ti and 40 wt. % Nb (Ti40Nb) [16]. This machine supports SLS/SLM synthesizing products of various physical configurations from a powdered material. The products can be sintered both in vacuum and in diverse gaseous mediums. The layout of the machine VARISKAF-100MB is given in Figure 4.

As the result of carried out experiments a set of samples is produced in the following process conditions. Laser emission power is 68, 75, 86, 96 and 106 W; scanning rate is varied in the range 0.017-0.05 m/s; scanning pitch is 0.1 mm.

Multilayer 10x10 samples are produced when thickness of powder layer is 0.7 mm. Samples are melted in argon. Plates made of alloy BT1-0B are used as a substrate. The substrate with powder is heated up to 200 °C, kept for 15 minutes in vacuum at the certain temperature, and melted in argon afterwards. The process temperature is controlled by a thermocouple of grade XA, the junction of which is in a substrate. Argon is supplied to the chamber as long as melting is carried out.



Figure 4. The layout of the machine VARISKAF-100MB: 1 – «flying» optical instruments; 2 – vacuum chamber; 3 – evacuation system; 4 – ytterbium laser; 5 – machine control assembly; 6 – computer numerical control.

An operating area, which shows preheating the substrate and melting the powder, is given in Figure 5.



Figure 5. Operating area of the machine.

Laser beam scanning strategy is a zigzag line (Figure 6).



Figure 6. Scanning strategy.

3. Results and Discussion

Optical images of Ti40Nb samples are given in Table 1. As one can see, increasing scanning rate results in augmentation of quantity and dimensions of pores. Samples made at laser power 68–106 W and scanning rates 0.05 and 0.042 m/s do not differ much. However, the quantity of pores is decreased and uniform areas of melted powder are formed as the rate of scanning is reduced to 0.017–0.042 m/s and emission power grows up to 96–106 W (Figure 7).



Figure 7. The relation of sample porosity to the laser emission power at various scanning rates.

The heat input grows and porosity decreases, as one can see in Figure 7, because of the increase in laser emission power and slowing down rate of scanning. Laser emission impact on the powder section takes longer as the rate of scanning is reduced, so fusion of the layer gets better.



 Table 1. Single layers of alloy Ti40Nb

We measure the formed layer thickness in samples produced under emission power 106 W. In Figure 8 one can see that penetration depth is reduced as the rate of scanning is decelerated.



Figure 8. The relation of the melted layer thickness to the rate of scanning.

As the consequence, we carried out an experiment and made a sample in the conditions: laser mission power 106 WBT; scanning rate 0.017 m/s and pitch of scanning 0.1 mm. To increase the heat input a titanium substrate is pre-heated up to 400 °C. The sample produced in conditions of the maximally possible heat input of the machine is shown in Figure 9.



Figure 9. A melted layer of a composite powder in conditions of a maximal heat input.

The porosity of the sample is approximately 9 %, however, the thickness of the melted layer increases considerably to 570 μ m, probably because of increasing temperature and the total heat input.

4. Conclusion

To sum up a conclusion can be drawn that laser emission power and rate of scanning are principal variables, which are important to form a melted layer of Ti40Nb composite powder. It should be noted

that the slowing down rate of scanning and the increasing laser emission power result in the decrease of porosity and increase in the thickness of the formed layer of the alloy.

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References

- [1] Sharkeev Y P, Eroshenko A Y, Kulyashova K S, Fortuna S V, Suvorov K A, Epple M, Prymak O, Sokolova V, Chernousova S 2012 Microstructure, mechanical and biological properties of zirconium alloyed with niobium after severe plastic deformation *J. Materialwissenschaft und Werkstofftechnik*, 44 (2-3) 198–204
- [2] Zhuravleva K, Scudino S, Khoshkhoo M S, Gebert A, Calin M, Schultz L, Eckert J 2013 J. Advanced Engineering Materials 4 262–268
- [3] Sharkeev Y P, Eroshenko A Y, Glukhov I A, Zhu Q, Tolmachev A I 2014 J. Physical Mesomechanics of Multilevel Systems 1623 567–570
- [4] Sharkeev Y P, Kovalevskaya Z G, Zhu Q, Khimich M A, Parilov E A 2015 Advanced Materials Research 1085 307–311
- [5] <u>http://dentaltechnic.info/index.php/implanty</u>
- [6] Kuznetsov V E 2003 J. CAD/CAM/CAE Observer 4 (13) 2–7
- [7] <u>http://dentaltechnic.info/index.php/implanty/zubnoe-protezyrovanye-na-ymplantatakh/2836-tyehnologiya_izgotovlyeniya_implanta</u>
- [8] Gibson I B, Rosen D W, Stucker B 2009 Additive manufacturing technologies. Rapid prototyping to direct digital manufacturing (New York, USA: Springer)
- [9] Shishkovsky I V, Zakiev E S, Kholpanov L P 2005 SLS layer-by-layer synthesizing products of titanium nitride *J. The Physics and Chemistry of Materials Processing* **3** 71–78
- [10] Saprykina N A, Saprykin A A 2012 Engineering support for improving quality of layer-by-layer laser sintering, *IFOST2012* 129–132
- [11] http://www.custompartnet.com/wu/selective-laser-sintering
- [12] Kumar S, Kruth J-P 2009 Composites by Rapid Prototyping Technology J. Materials & Design 1–23
- [13] Ibragimov E A, Saprykin A A, Babakova E V 2014 Influence of Laser Beam Machining Strategy at SLS Synthesis, *Advanced Materials Research* **1040** 764–767
- [14] Murali K, Chatterjee A N, Saha P, Palai R, Kumar S, Roy S K, Mishra P K, Choudhury A R 2003 Direct selective laser sintering of iron-graphite powder mixture J. Journal of Materials Processing Technology 136 179–185
- [15] Saprykin A A, Gradoboyev A V, Yakovlev V I, Ibragimov E A, Babakova E V 2015 Comparative analysis of processes to activate copper powder ΠMC-1 for SLS synthesizing products J. Metal processing 3 (68) 82–88
- [16] Saprykin A A, Ibragimov E A, Babakova E V and Yakovlev V I 2015 Influence of mechanical activation of copper powder on physicomechanical changes in selective laser sintering products, *AIP Conf. Proc.* 1683 020199
- [17] Saprykin A A, Ibragimov E A, Babakova E V 2015 Engineering and Technology Based on Rapid Prototyping J. Applied Mechanics and Materials **770** 622–627
- [18] Fischera M, Joguetb D, Robina G, Peltierc L, Laheurte P 2016 In situ elaboration of a binary Ti-26Nb alloy by selective laser melting of elemental titanium and niobium mixed powders. *J. Materials Science and Engineering* 62 852–859