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EBSD Examination of Argon Ion Bombarded Ti-6Al-4V Samples Produced with DMLS Technology

Zoltan Keresztes[1*], David Pammer[1], Janos Peter Szabo[1]

[1] Department of Materials Science and Engineering, Faculty of Mechanical Engineering, Budapest University of Technology and Economics, H-1111 Budapest, Műegyetem rkp. 3, Hungary
[*] Corresponding author, e-mail: kzoltan@eik.bme.hu

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Abstract

Additive manufacturing (AM) indicated great technological increase in the last years. Primarily development of new methods, new computer softwares and more useable materials are responsible for this progression. AM has numerous advantages that cause the appearance of it in almost every field of industry. However this widely spread technology has many under examined properties, especially those, that uses metal as raw material. One of these less understood properties is the behavior of grains during the melting phase and the microstructure after production. The main aim of this study is to examine the microstructure of ion bombarded – using argon - AM produced Ti-6Al-4V samples applying EBDS investigation, measuring the grain size, and the orientation of the grains.

Keywords

additive manufacturing, DMLS, ion bombardment, EBSD, grain size, misorientation

1 Introduction

Additive manufacturing is a rapidly developing technology which can be found almost in every field of industry and beyond. The first step of this kind of production is always a well prepared model using some sort of CAD program. Then an AM device starts to build this model from layer-by-layer according to the given model geometry. There are numerous variations of this technology depending on the raw material, the energy source that make contact between the layers, etc. [1]. The main advantages of AM – comparing to other manufacturing – are to customized much more freely the product, to prepare more difficult, complex and heterogeneous geometries, using less specialized tools, significantly reducing production time and costs, while also decreasing postproduction time and steps [2].

Mainly the improvement of laser energy sources causes continuously increasing number of usable raw materials. Titanium alloys take special place in these materials, because in a traditional way, the production is quite expensive and difficult, especially in biomedical engineering, where very complex and customized geometries are needed.

There are some articles, which are investigating the microstructure, the morphology of AM produced titanium alloys with special grinding, polishing and etching techniques. This paper is examining also the microstructure and the morphology of titanium alloy samples produced with an AM technology – it is a powder bed type one, called DMLS. This publication is a summary of the examinations that were performed on Ti-6Al-4V titanium alloy. The samples were produced with direct metal laser sintering (DMLS) technology. After production, we prepared the specimens for metallographic investigation. The samples were grinded, polished, surface etched – using Keller's reagent – and treated with a special technology, so called ion bombardment. We examined the homogeneity of the surface, perpendicular to the sintering laser. The examinations contain the measurement of the grain sizes and the misorientation of the grains with the help of EBSD.

2 Material and methods

2.1 Raw material

Nowadays more and more metals and metallic alloys take place in AM as raw material owing to the continuous developments. This study represents the examinations and results that were performed using Ti-6Al-4V samples. This kind of titanium alloy has ideal strength/density rate and first of all it is biocompatible. These properties make Ti-6Al-4V to be used widely in biomedical engineering.

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The chemical composition of this alloy rather has intervals, than exact values (Table 1) [3].

2.2 DMLS

Direct Metal Laser Sintering (DMLS) is a special form of Powder Bed Fusion AM technology which use laser to sinter the raw material layer by layer [5]. Fig. 1 shows the schematic illustration of DMLS process [6].

Fig. 2 shows the three main part of a layer. The raw material coated the building platform. After the coating the laser starts to sinter the powder bed according to the geometry. When the laser finished the sintering process, than one layer is ready. The table is lowered by a given layer thickness and the powder bed coating starts again and followed by the sintering [7] The samples were produced with the original process parameters, which was developed by the manufacturer. The produced samples were not post processed and heat treated.

2.3 EBSD and ion bombardment

Electron Backscatter Diffraction (EBSD) in one the most interesting and most used accessory system that can be attached to Scanning Electron Microscopy (SEM). The bases of this technology can be linked to Shoji Nishikawa and Seishi Kikuchi who were investigated fundamental diffractions in 1928 [8].

Microstructure is a link between the science and the technology of materials. Although scientists usually think about microstructure primarily in terms of grain morphology, EBSD means much more. Complete microstructure analysis is provided with the following:

- grain structure (size/distribution)
- grain boundary characteristics
- macro and micro –crystallographic texture
- phase discrimination and distribution
- deformation
- crystal orientation
- epitaxy between layers
- etc…[9]

EBSD operates by arranging a flat, highly polished sample at a shallow angle - usually 20° (or 70° comparing to SEM stage) – to the incident electron beam. Electron diffraction occurs from the incident beam point on the sample surface. When the beam stationary, an EBSD pattern emanates spherically from this point. When the primary beam interacts with the crystal lattice, then low energy loss backscattered electrons are channelled and are subjects to path differences which cause constructive and destructive interference. When a phosphor screen is placed near to the titled sample, in the path of the diffracted electrons, a diffraction pattern can be seen [8, 10]. During the experiments, that are shown in this study FEI Quanta 3D SEM was used equipped with Hikari – camera for EBSD investigations.

Table 1 Chemical composition of Ti-6Al-4V material [4]

Elements	Percentage Wt (%)
Titanium (Ti)	88.1 – 91
Aluminum (Al)	5.5 – 6.75
Vanadium (V)	3.5 – 4.5
Iron	<0.3
Oxygen	<0.2
Carbon	<0.1
Nitrogen	<0.05
Hydrogen	<0.015

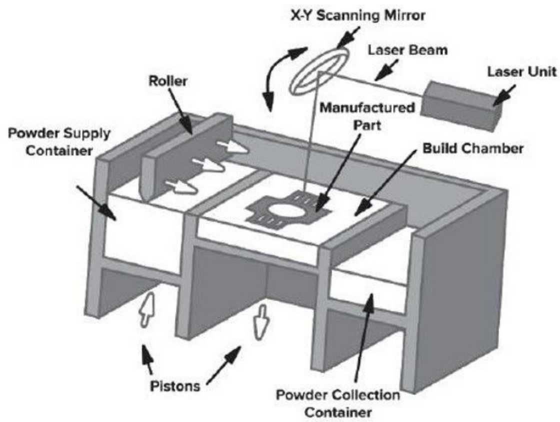

Fig. 1 Schematical illustration of DMLS process

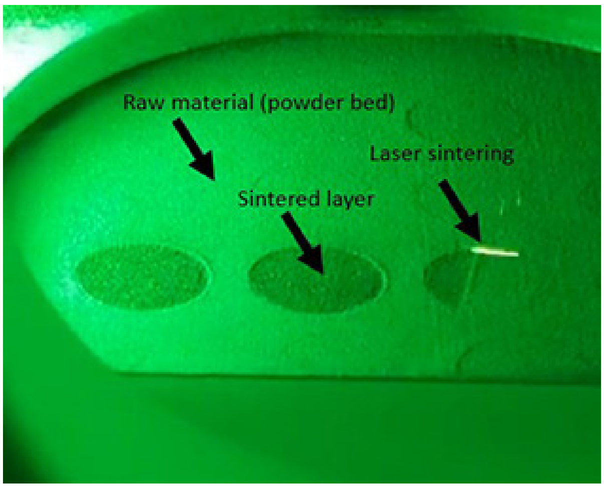

Fig. 2 DMLS technology with raw material, sitered layer and the laser sintering

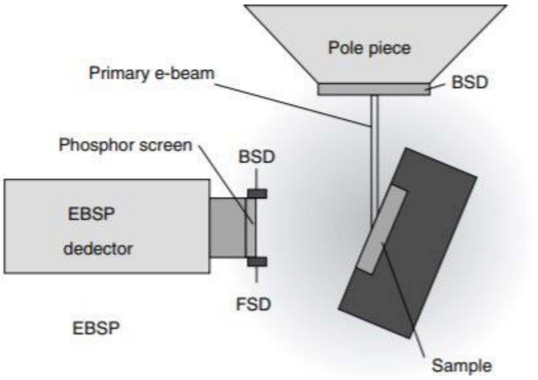

Fig. 3 Schematic arrangement of sample orientation in the SEM

As always the first step of a useful examination is the precise sample preparation. In this study Ti-6Al-4V specimens were leaded up to EBSD investigation. After the resin fixing and grinding, the samples were polished with 1 µm grain sized Silica disk. For better preparation – in parallel gaining better EBSD results – Ar ion bombardment was applied. Ion bombardment is a process often used in several manufacturing steps of very large and ultralarge scale integration circuits, such as ion implanting and dry etching, as well as in substrate sputter cleaning carried out before other processes. Nowadays the application of ion beam is considered as one of the most effective approaches to improve the surface morphology [11].

3 Results

After polishing and Ar ion bombardment the chemical composition was specified in different areas of the sample. The cylinder sample – with h = 6mm, d = 4mm – was divided for 5 zones along the diameter. Table 2 shows the average main values of the measured compositions in the different zones compared with the traditional chemical composition of Ti-6Al-4V according to Table 1.

EBSD investigation resulted information about two different zones of the sample surface. The zones take places in the center and in the edge of the specimen where the laser most likely causes different grain structures. Therefore inhomogeneous surface is expected. Fig. 4 shows the outer edge of the specimen where green box signs the examination area. Fig. 5 shows the result of EBSD examination including image quality (IQ) inverse pole figure (IPF) picture and the mixture of them.

DMLS technology often results heterogeneous grain structure due to the difficult processes during melting and sintering phases. Examining the inner parts of the sample not just provides more information about the grains and phases, but homogeneity of them. SEM picture was taken of the central zone and the green box also signs the examined area by EBSD (Fig. 6).

With the help of EBSD IQ, IPF and IPF on IQ pictures were taken again, which are shown in Fig. 7.

As EBSD images shows, both the inner and the outer zone has quite the same microstructure. Despite of the similarity in microstructure, there is a heterogeneous area in center zone signed with pink color in IPF. To determine the homogeneity of the microstructure in the outer and inner zone we measured the grain size and the misorientation of the grains. Fig. 8 and Fig. 9 summarize the grain size and the misorientation of the examined area in the edge of the specimen.

After measuring the misorientation and grain size of the outer area, we also examined the inner region to get comparable information. Fig. 10 and Fig. 11 contain these values.

Table 2 Chemical composition of DMLS produced Ti-6Al-4V sample compared to the traditional composition

Elements	Base Wt (%)	Measured average Wt (%)
Titanium (Ti)	88.1 – 91	91.44
Aluminum (Al)	5.5 – 6.75	6.01
Vanadium (V)	3.5 – 4.5	2.8

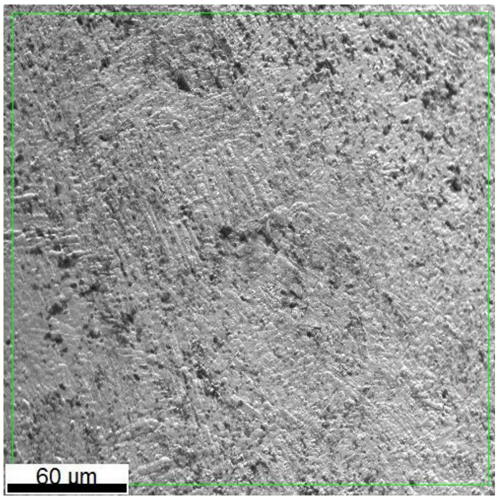

Fig. 4 SEM picture of the outer zone of the specimen

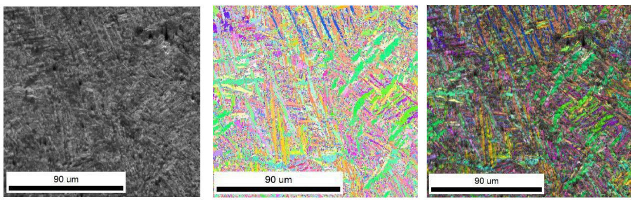

Fig. 5 IQ (left), IPF (center) and IPF on ID (right) pictures of the edge

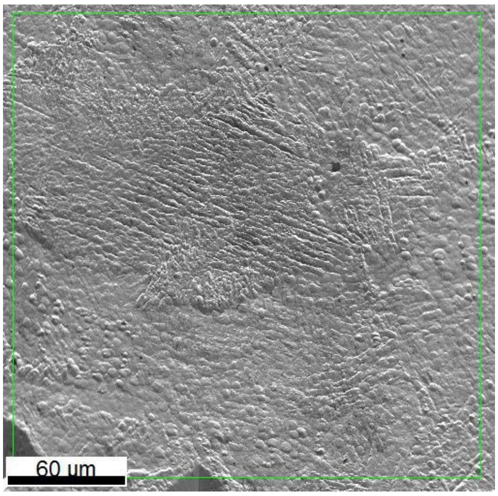

Fig. 6 SEM picture of the center zone of the specimen with the signed investigation area

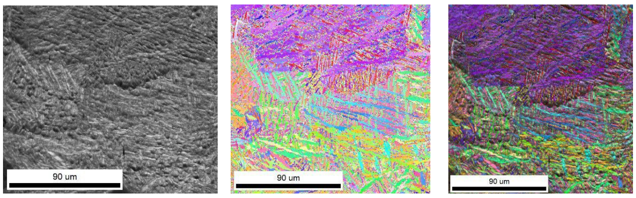

Fig. 7 IQ (left), IPF (center) and IPF on IQ (right) pictures of the center zone)

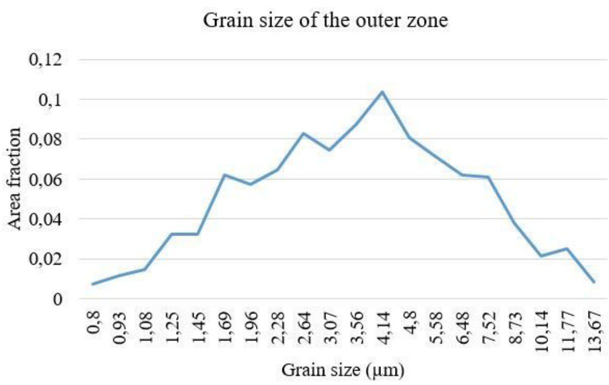

Fig. 8 Grain size of the outer zone

As it can be detected in Fig. 5 and Fig. 7, both have needle microstructure with almost the same sized and orientated grains. Examined more closely, in Fig. 7 there is homogeneous area – signed with pink color - which either a separated, inhomogeneous grain, or a special area, filled with also the same orientated and sized grains. We specifically measured the grain size and the misorientation of this region, Fig. 13 and Fig. 14 show the results.

The sizes and orientations of the measured grains in the center and the edge of the specimen is homogeneous such

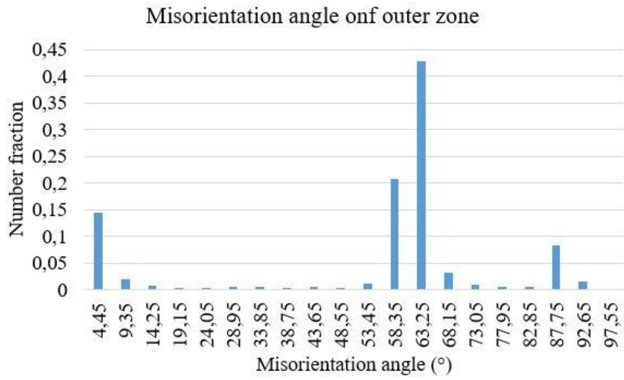

Fig. 9 Misorientation of outer zone

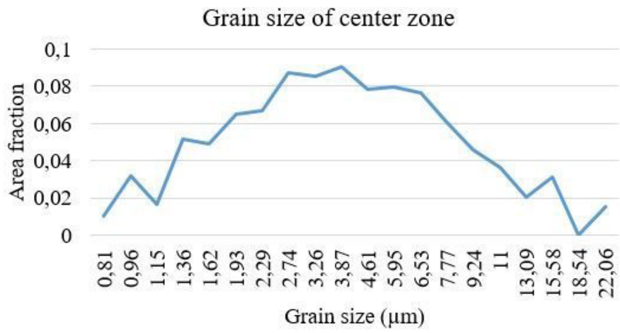

Fig. 10 Grain size of center zone

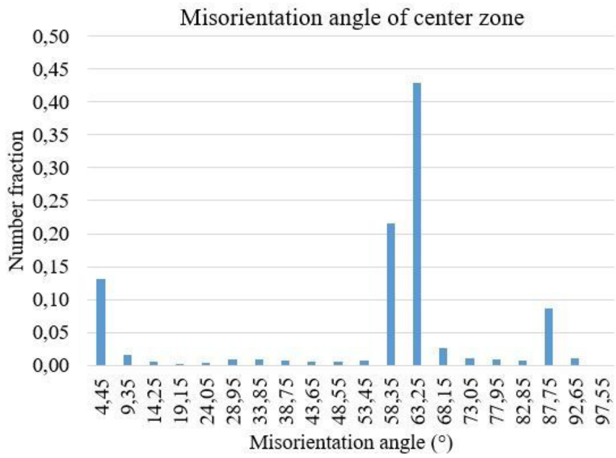

Fig. 11 Misorientation of center zone

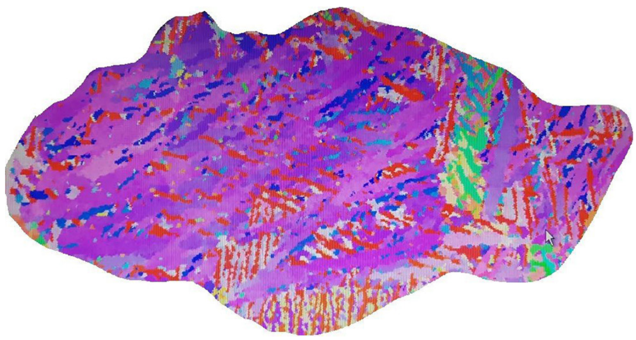

Fig. 12 Specific, inhomogeneous area cut off the IPF picture

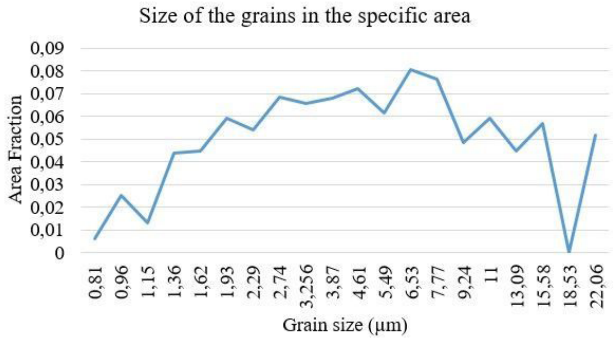

Fig. 13 Grain size of specific area

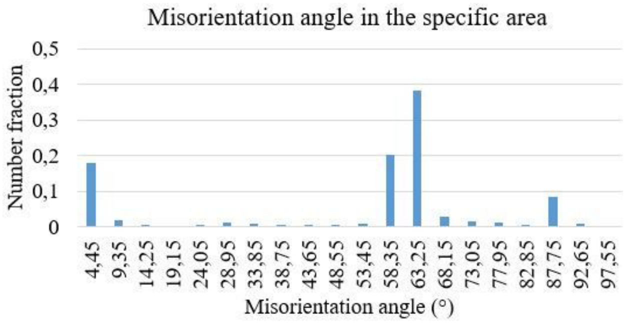

Fig. 14 Size of the grains in the specific area

as these values examined in the specific area signed in IPF picture. Table 3 and Table 4 contains the hugest, smallest and the average grain sizes and misorientations about the three examined region.

Table 3 Grain sizes of the different areas and the average grain sizes

Zone/size (μm)	Smallest grain	Largest grain	Average grain size
Center zone	0.81	22.06	1.91
Edge of specimen	0.80	13.67	2.02
Specific area	0.81	22.06	2.03

Table 4 Misorientation of the grains in the different area and the average misorientation angles

Zone/misorientation angle (°)	Smallest angle	Largest angle	Average angle
Center zone	4.45	92.65	55.14
Edge of the specimen	4.45	92.65	54.39
Specific area	4.45	92.65	51.93

4 Conclusion

This paper shows investigation of Ti-6Al-4V alloy microstructure – produced with DMLS technology - using EBSD. Well-prepared samples, using polishing, surface etching and Ar ion bombardment were key factors for the examination. Applying 3D printing technologies the main aim is to produce workpieces from virtual models without involving any hybrid technology or using post production method. Nowadays this aim is quite far from the current condition of 3D printing, but the development of raw materials, technologies will surely make this possible.

Sintering the raw material in the way of creating a homogeneous, definite structure makes 3D printing closer to this aim. Our results show that the average grain size in two different zones of the specimen is nearly the same (1.91 μm – center zone, 2.02 μm – edge of the specimen). The average orientation of these grains is also nearly the same (55.14° - center zone, 54.39° - edge of the specimen).

Examining the center zone of the specimen image quality (IQ) and inverse pole figure (IPF) show a heterogeneous region. Grain size and misorientation were defined in this region to compare the values with the homogeneous zone. The average grain size is 2.03 μm and the average misorientation angle is 51.39° in the inhomogeneous region. So these values are almost meeting the homogeneous datas.

In the future a whole new investigation will be acted, where the examined surface is parallel with the sintering laser, not perpendicular. This change will let us examining the homogeneity of the grains in the building direction and between the layers.

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References

[1] Gibson, I., Rosen, D. W., Stucker, B. "Additive Manufacturing Technologies", Springer, New York, 2009.

[2] Rejeski, D., Zhao, F., Huang, Y. "Research needs and recommendations on environmental implications of additive manufacturing", Additive Manufacturing, 19, pp. 21–28, 2018. https://doi.org/10.1016/j.addma.2017.10.019

[3] Mandal, V., Hussain, M., Kumar, V., Kumar Das, A., Singh, N. K. "Development of reinforced TiN-SS316 metal matrix composite (MMC) using direct Metal laser sintering (DMLS) and its characterization", Materials Today, 4(9), pp. 9982–9986, 2017. https://doi.org/10.1016/j.matpr.2017.06.306

[4] EOS GmbH - Electro Optical Systems "EOS Material data sheet, EOS Titanium Ti64", [online] Available at: https://cdn.eos.info/a4eeb73865d54434/5926811b3739/Ti-Ti64_9011-0014_9011-0039_M290_Material_data_sheet_11-17_en.pdf [Accessed: 08 July 2018]

[5] ACF Group "Laser Sintering", [online] Available at: https://www.advancedcustomfields.com/resources/group/ [Accessed: 08 July 2018]

[6] Maitland, T., Sitzman, S. "Electron Backscatter Diffraction (EBSD) Technique and Materials Characterization Examples", In: Zhou, W., Wang, Z. L. (eds.) Scanning Microscopy for Nanotechnology, Springer, New York, NY, USA, 2006. https://doi.org/10.1007/978-0-387-39620-0_2

[7] Dutta, B., Froes, F. H. "The Additive Manufacturing (AM) of titanium alloys", Metal Powder Report, 72(2), pp. 96–106, 2017. https://doi.org/10.1016/j.mprp.2016.12.062

[8] Schwartz, A. J., Kumar, M., Adams, B. L., Field, D. P. (eds.) "Electron Backscatter Diffraction in Materials Science", Kluwer, New York, NY, USA, 2000.

[9] Zhou, W., Wang, Z. L. (eds.) "Scanning Microscopy for Nanotechnology, Techniques and Applications", Springer, New York, NY, USA, 2007. https://doi.org/10.1007/978-0-387-39620-0

[10] Marqués, L. A., Jaraiz, M., Rubio, J. E., Vicente, J., Bailón, L. A., Barbolla, J. "Molecular dynamics simulations of ion bombardment processes", Materials Science and Technology, 13(11), pp. 893–896, 1997. https://doi.org/10.1179/mst.1997.13.11.893

[11] Ramezani, A. H., Sari, A. H., Shokouhy, A. "The effects of argon ion bombardment on the corrosion resistance of tantalum", International Nano Letters, 7(1), pp. 51–57, 2017. https://doi.org/10.1007/s40089-017-0201-7