Journal of the Technical University of Gabrovo

https://mc04.manuscriptcentral.com/jtug

INVESTIGATION OF HARDNESS AND ELASTIC MODULUS OF MILLED AND SLM Ti6Al4V ALLOY

Tsanka Dikova[1*], Vladimir Dunchev[2], Yavor Gagov[1]

[1]*Medical University of Varna, 9000 Varna, Bulgaria*

[2]*Technical University of Gabrovo, 5300 Gabrovo, Bulgaria*

ARTICLE INFO

Article history:
Received 17 October 2022
Accepted 3 November 2022

Keywords:
Ti6Al4V, CAD/CAM milling, selective laser melting, hardness, modulus of elasticity

ABSTRACT

The aim of the present paper is to investigate the hardness and elastic modulus of Ti6Al4V alloy produced by CAD/CAM milling and selective laser melting. The Vickers micro-hardness is measured, while the modulus of elasticity is determined by new developed methodology consisting of bending test and finite element analysis. It was established that the manufacturing methodlead to differences in the micro-hardness and elastic modulus of the milled and SLM samples. The measured micro-hardness of the Ti6Al4V alloy was higher for the SLM samples (396 HV) compared to the milled ones (347 HV). The values of the modulus of elasticity of Ti6Al4V alloy are 180 GPa for the milled and 120 GPa for the SLM fabricated samples. They are higher than the data given in the literature or by the manufacturers. Subsequent heat treatment with porcelain firing modes slightly decreased the micro-hardness to 322 HV for the milled and 388 HV for the SLM produced samples and had almost no effect on the modulus of elasticity for both groups.

1. INTRODUCTION

Pure titanium and its alloys are characterized by low relative mass, good mechanical properties, high corrosion resistance and biocompatibility. In addition, the modulus of elasticity of titanium is close to that of the natural bone. For these reasons, Ti and titanium alloys are widely used in implantology for fabrication of various types of implants [1-3]. In recent years, their application has been extended to other areas - prosthetic dentistry. Due to its low relative mass, Ti and its alloys are suitable for production of infrastructures for metal-ceramic prosthetic constructions - crowns, bridges and over-implant prostheses [4, 5].

Lost wax casting was introduced in dentistry and dental laboratory at the beginning of the last century [6]. Until ten years ago, it was the main technology for production of dental constructions. Titanium and titanium alloys are characterized by high melting temperatures (1668ºC for pure Ti). In addition, titanium is a highly reactive metal, and in order to obtain quality castings, it is necessary to carry out the casting process under special conditions - vacuum or a protective atmosphere [7]. All this complicates the casting process, makes it difficult and expensive to produce details from Ti and its alloys.

Modern CAD/CAM technologies are a successful alternative for manufacturing metal infrastructures for dental prostheses from Ti and titanium alloys [5,7,8]. When using CAD/CAM systems, the virtual model of the detailis generated by the CAD module using specialized software. The actual objectis made from a variety of materials using different technological processes depending on the type of machine representing the CAM module. The CAM module equipment, that is used to produce dental constructions, most often includes a milling machine or a 3D printer. The milling machine is commonly used for manufacturing prosthetic restorations from zirconia and other types of ceramics, and recently also for infrastructures from dental alloys – cobalt-chromium, nickel-chromium or titanium. In this process, the detailis producedfrom a solid blank with guaranteed mechanical properties. Therefore, the mechanical properties of the construction are not expected to change.

3D printers work on the principle of building the object layer by layer [8]. This technology is emerged in the 1980s and has undergone rapid development in the last ten years. New technological processes are constantly being developed, and corresponding new materials are created for them. Practically all known groups of materials can be used in 3D printing technologies – metals and alloys, polymers and composites, ceramics and gypsum. When working with metals and alloys, the process of selective laser melting (SLM) is used.In this process, powder of pure metal or alloy is used as starting material, which is spread on the table of the machine in a layer of definite thickness [5,7,8]. The virtual model of the detailis pre-cut into layers of the same thickness. After that, a laser beam is used to scan the section of the detail from the corresponding layer. In the same way, the section of the part from the next layer is melted on the previous layer. Thus, by sequentially welding layer upon layer, the entire detail is built [8-12]. As a result

[*] Corresponding author. E-mail: tsanka.dikova@mu-varna.bg

http://doi.org/10.62853/ZECM7737

of the rapid heating and cooling in the SLM process, the microstructure of the manufactured detail is fine and relatively more homogeneous on the one hand, and on the other hand, high residual stresses are generated in it. All this leads to higher hardness and mechanical properties of the SLM alloy [11-15]. For stress relaxation, subsequent heat treatment is applied, which leads to changes in the microstructure, an increase in relative elongation, a decrease in hardness and anisotropy [13, 15-17].

Zhang XY et al. [13] obtained a hardness of 365 HV on a SLM manufactured Ti6Al4V alloy. After heat treatment at a temperature of 800ºC, it is reduced to 330-363 HV depending on whether the cooling is in furnace or air respectively. According to Liu Z et al. [14] the micro-hardness of the SLM Ti6Al4V alloy after annealing at a temperature of 930ºC is 297 HV. It increases with increasing the temperature and reaches 318 HV at 980ºC.

The two technologies - milling and SLM, used for manufacturing dental constructions from the CAM module, fundamentally differ in the method of production. This leads to differences in the microstructure and mechanical properties of the resulting details. Therefore, the aim of this paper is to investigate the hardness and elastic modulus of Ti6Al4V alloy produced by CAD/CAM milling and selective laser melting. The modulus of elasticity is determined by new developed methodology consisting of bending test and finite element analysis.

2. MATERIALS AND METHODS

2.1. Materials and methods for samples manufacturing

Two groups of samples (6 pieces in each) were made from Ti6Al4V alloy by means of CAD/CAM milling and selective laser melting. The samples were plate-shaped with the following dimensions: 25 mm x 3 mm x 0.5 mm. For their manufacture, a virtual model was initially generated using SolidWorks software, which was then converted into stl-format. The same virtual model was used for the production of the samples by both technologies.

- CAD/CAM milling

The specimens are fabricated from Starbond Ti5 Disc titanium alloy (Ti6Al4V milling Grade 5 “ELI”) for ISO 22674 type 4 dental restorations with chemical composition given in Table 1 [18]. The workpiece used is disk with a thickness of 10 mm and a diameter of 98.3 mm, Ref.136510. The location of the samples during milling is shown in Fig.1a. The plates are milled on a CORITEC 650i Loader machine (Imes-Icore GmbH, Eiterfeld, Germany).

Table 1 *Chemical composition ofTi6Al4Valloy (milling disc - 1 and powder for SLM - 2) [18,19]*

Chemical element, (%) → Alloy ↓		Al	C	H	Fe	N	O	Ti	V
1	Starbond Ti5 Disc	6.2	<0.4	<0.4	<0.4	<0.4	<0.4	89.4	4
2	CT PowderRange Ti64 F	5.50-6.50	0.08	0.012	0.25	0.05	0.13	Rest	4.50

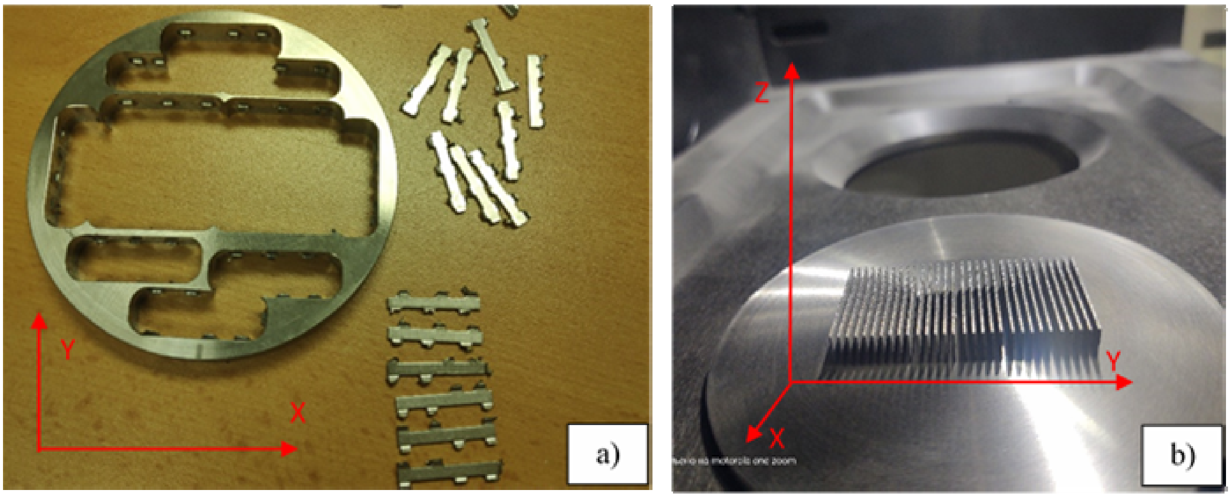

Fig. 1. Ti6Al4V alloy disc with milled samples – a) and disposition of the samples on the working table of SLM machine – b)

- Selective laser melting

CT PowderRange Ti64 F (Ti6Al4V) alloy with chemical composition, given in Table 1 [19, 20], from the manufacturer Carpenter Additive (Liverpool, UK) is used as starting material for the samples made by SLM. The powder alloy is characterized with an average particle size of 15-45 µm, with 5% being smaller than 15 µm and 7% being larger than 45 µm. The main part of the fraction consists of particles with sizes of 18-24 µm, and the rest - between 32.5-34.4 µm. The samples were made by the 3D MEDICAL PRINT company (Pleven, Bulgaria) on a SYSMA MySint 100 machine (SYSMA S.p.A., Vicenza, Italy), equipped with a fiber laser. The following parameters of the SLM process were used: laser power 200W, laser beam spot diameter 55 µm, layer thickness 20 µm, scanning step 0.020 mm, and argon as protective gas. The placement of the specimens and supports during SLM is shown in Fig.1b. More details about the fabrication of the samples are given in [21]. After their fabrication, the samples were subjected to isothermal annealing to relieve internal stresses. It was carried out in a protective environment of argon. It consisted of heating and holding the samples at a temperature of 600°C for 30 min, subsequent heating to temperature 800°C with holding time of 1h 40 min and cooling with the furnace.

- Heat treatment

Three samples from each group were subjected to additional heat treatment, imitating sequential firing of 4 layers of dental porcelain. The modes are given in Table 2 and correspond to the firing of the opaque layer, 2 dentin layers and a glaze of VITA LUMEX AC ceramics (VITA Zahnfabrik H. Rauter GmbH & Co.KG, Bad Sackingen, Germany).

***Table 2** Regimes of heat treatment, simulating porcelain firing [22]*

№	Heating	Preheating, °C	Hold. time, min	Tempera-ture increase, °C/min	Max tempera-ture, °C	Hold. time, min	Cooling to °C
1	1st heating (opaque firing)	400	4	50	800	1	
2	2nd heating (first dentine firing)	400	6	50	760	1	500*
3	3rd heating (second dentine firing)	400	6	50	755	1	500*
4	4th heating (glaze firing)	400	0	80	750	1	500*
* Long-term cooling down to the appropriate temperature							

2.2. Characterization of the samples

- Micro-hardness investigation

The micro-hardness of milled and SLM samples before and after heat treatment mimicking the porcelain firing was investigated. The hardness measurement was carried out by the Vickers method on grinded surfaces of the specimens. Five measurements per sample were made with a ZHVµ-S micro-hardness tester (Zwick/Roell, Germany), applying a load of 25 g for 10 s. The arithmetic mean value was used in the analysis.

- Determining modulus of elasticity

A new combined methodology has been developed to define the Young's modulus, including a three-point bending experiment and subsequent finite element analysis (FEA) of the test.

The experiment represents bending of a beam supported on rounded tip supports (1 mm radius) and loaded with a rounded tip punch (1 mm radius). The beam is bent in the middle up to 0.3 mm, in order to preserve the linear relationship betweenthe force and displacement (Fig. 2), thus ensuring loading only in the elastic region. Three samples of the milled and SLM-made Ti6Al4V alloy were subjected to bending with a ZWICK RoellVibrophore 100 machine (ZWICK Roell, Germany) before and after heat treatment according to the modes, imitating the production of a porcelain coating.

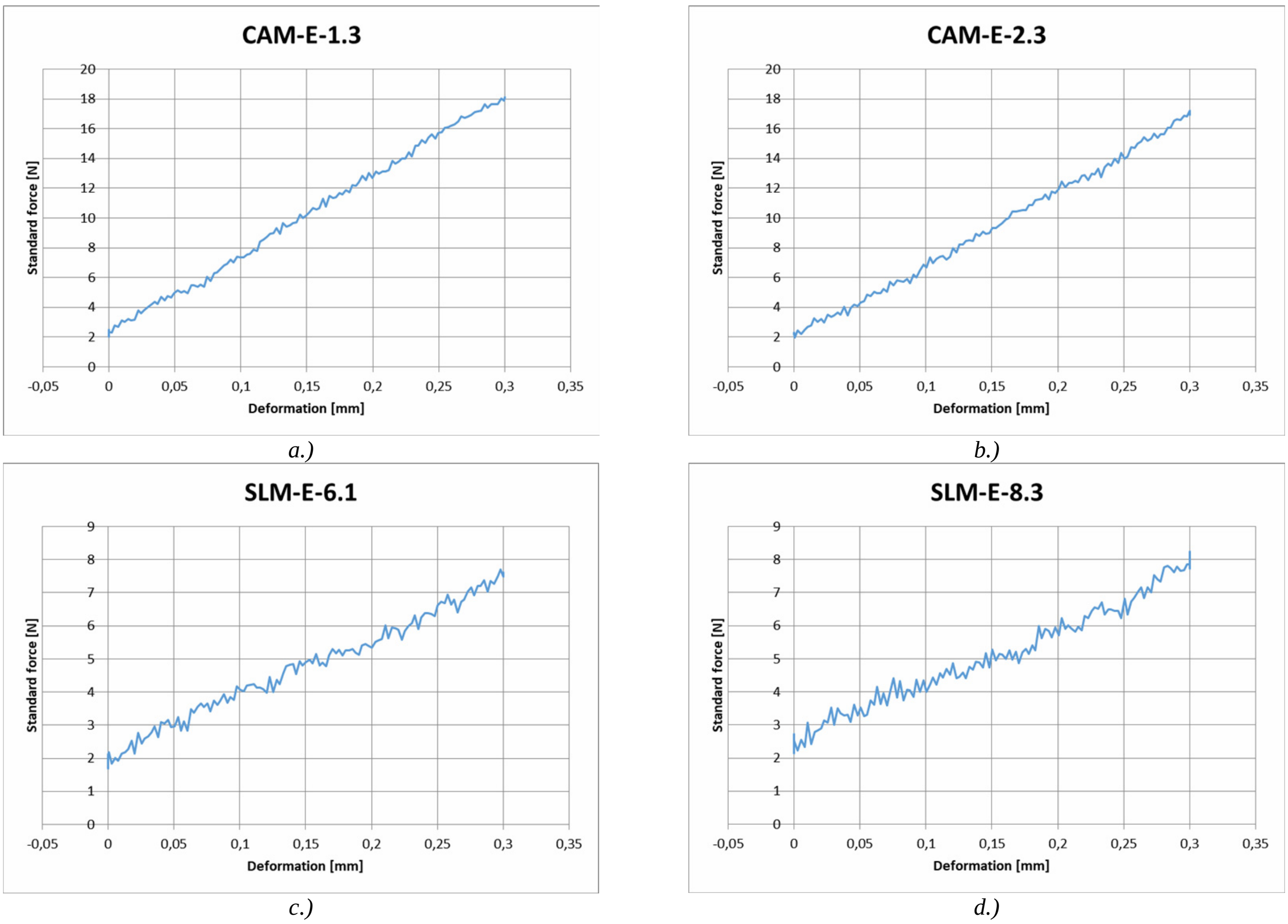

Fig. 2. Bending stress-strain plots of titanium alloy specimens used to determine modulus of elasticity.Milled alloy before – a) and after – b) heat treatment and SLM alloy before – c) and after – d) heat treatment

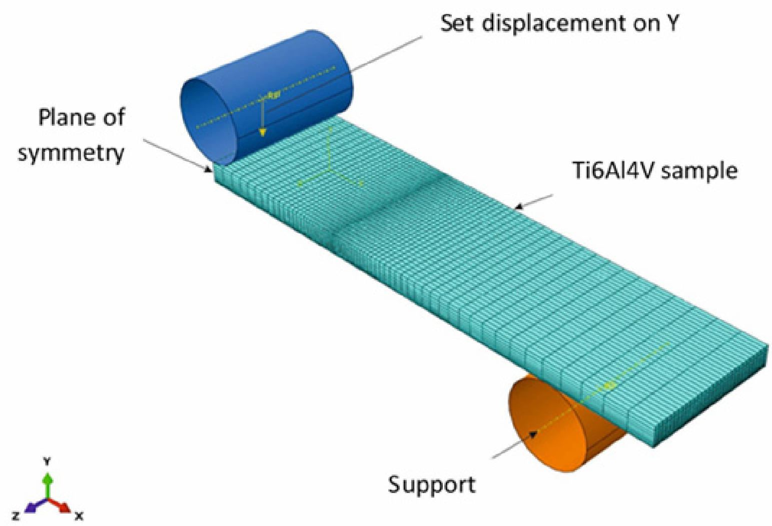

Fig. 3. FE model for determining the modulus of linear deformations of TiAl6V4 alloy

The linear FEA was carried out on specialized software ABAQUS. The virtual model consists of three bodies - beam, punch and support. In order to minimize the computational time and due to the axis of symmetry of the conducted test, a FEA model is used only on half of the computational scheme (Fig. 3). The beam is modeled as a rigid deformable body with the absolute dimensions of the plate from the experiment. The punch and one support are designed as a cylindrical ideal rigid body with a base radius of 1 mm. The displacement time in the constructed model is equal to 0.3 s, in order to be able to account for the force accurately for the entire duration of the load. An iterative approach was applied to determine the Young's modulus. A Poisson's ratio of 0.33 was assumed, and the magnitude of the Young's modulus was varied to match the force reported in the experimental test (Fig. 2) and the finite element model.

3. RESULTS OBTAINED

- Micro-hardness

The measured micro-hardness of the Ti6Al4V alloy was 347 HV for the milled specimens and 396 HV for the SLM ones (Fig. 4). It is very close to the one given by the manufacturer – 330 HV and 405 HV respectively [18-20]. After heat treatment with the porcelain firing modes, the micro-hardness of both groups of samples decreased slightly to 322 HV for the milled and 388 HV for the SLM produced.

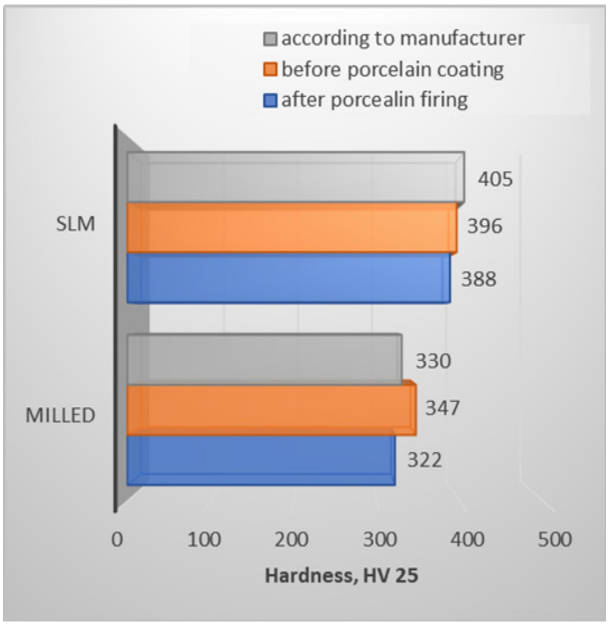

Fig. 4. Micro-hardness of Ti6Al4V alloy

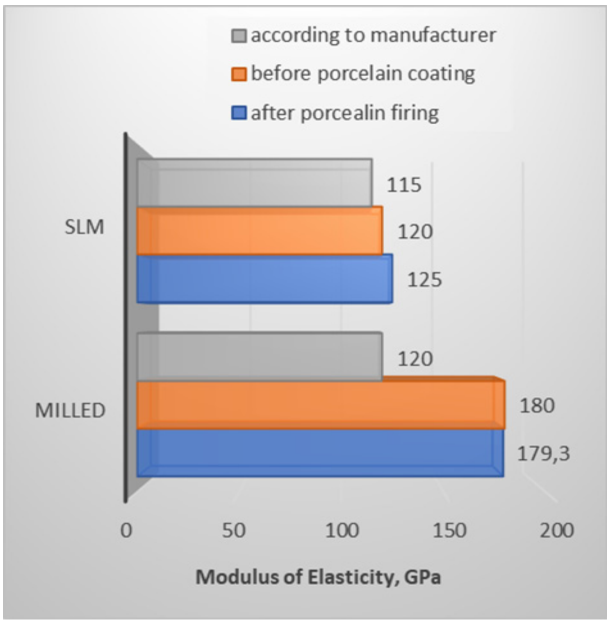

Fig. 5. Modulus of elasticity of Ti6Al4V alloy

- Modulus of elasticity

The values of the modulus of elasticity of the Ti6Al4V alloy, determined by the combined methodology, are 180 GPafor the milled and 120 GPa for the SLM samples (Fig. 5). Heat treatment, simulating the porcelain firing, hardly changes the modulus of elasticity.It is 179.3 GPa for the milled specimens and 125 GPa for the SLM fabricated ones. They are higher than the data given in the literature (110-136 GPa for milling alloy) [7, 9] or by the manufacturers (110-115 GPa for the SLM alloy) [19].

4. DISCUSSION

It is known that the production method determines the microstructure and mechanical properties of a given alloy.

The process of selective laser melting is characterized by rapid heating until melting of a layer of metal powder, its deposition on the previous layer and rapid cooling. As a result, on the one hand, a very fine, lamellar microstructure is obtained, and on the other hand, high residual stresses are generated. In addition, the already welded layers of the part are repeatedly heated so that near-surface temperatures may exceed those of phase transitions.

Ti6Al4V alloy is a representative of the group of titanium alloys with a two-phase α+β microstructure [14]. Due to the peculiarities of the process, the microstructure of Ti6Al4V alloy, produced by SLM, consists of acicular α′-martensite dispersed in the primary columnar grains of the β-phase [13-15]. This structure is responsible for the higher hardness, yield strength and tensile strength of the SLM alloy compared to the two-phase α+β Ti6Al4V alloy produced by conventional technologies. Heat treatment is applied to transform the microstructure containing α′-martensite to a two-phase α+β structure. It was established that the dissolution of α′-martensite and its transformation into an α+β structure begins already at temperatures above 400 °C [23], and the residual stresses relax completely after holding for 2 h at a temperature of 730°C [13]. At this temperature, the initial martensite transforms into lamellae of α+β phases. As the temperature increases to 800ºC, the initial acicular microstructure gradually transforms into a lamellar α+β structure. Increasing the temperature to 850ºC leads to a slight increase in the width of the α and β lamellae. Other authors also found that after heat treatment at temperatures of 780-800ºC, the martensitic structure transforms into a mixture of α+β phases, with the α phase existing in the form of lamellae [16,17]. A further increase in temperature to 950-980ºC leads not only to an increase in the thickness of the α and β lamellae and overall enlargement of the microstructure, but also to a decrease in the amount of the α phase (23% at 950ºC) [13]. These changes in the microstructure determine an increase in relative elongation and plasticity, as well as a decrease in hardness, strength and anisotropy [13,15-17].

In our study, a higher micro-hardness of the SLM samples (396 HV) of Ti6Al4V alloy was obtained compared to the milled ones (347 HV). These values are higher than the data of other researchers, but are close to the limits given by the manufacturers (405 HV and 330 HV respectively). From the overview made above, it can be seen that the main reason for the higher micro-hardness of the SLM samples compared to the milled ones is their specific microstructure. Annealing at 800ºC leads to release of the internal stresses, but also to an increase in the thickness of the α and β lamellae, which is the reason for the slightly lower micro-hardness of the SLM samples compared to that of the manufacturer. Since the additional heat treatment was carried out in the temperature range 750-800ºC, i.e. below the temperature of phase transformations, the slight decrease in hardness in both groups of alloys is most likely due to additional enlargement of the microstructure.

The modulus of elasticity of Ti6Al4V alloy produced by SLM (120 GPa) is slightly higher than the maximum given by the manufacturer, 115 GPa [19]. For the milled alloy, the modulus of elasticity is about 25% higher than the data in the literature (136 GPa) [7]. Since the modulus of elasticity was determined by a newly developed methodology based on a bending experiment of plates with a thickness of 0.5 mm, the most likely reasons for these differences are the scale factor and the difference in the determination methodologies. For the milled alloy, the placement of the specimens in the milling disc (Fig. 1a) should also exert an effect, taking into account the anisotropy of the mechanical properties of the workpiece. Subsequent heat treatment at temperatures below the phase transitions hardly changed the values of the modulus of elasticity for both groups of samples.

5. CONCLUSIONS

In the present work, the micro-hardness and elastic modulus of Ti6Al4V alloy, produced by milling and selective laser melting, were investigated. The manufacturing method, defining a specific microstructure of the Ti6Al4V alloy, leads to differences in the micro-hardness and elastic modulus of the milled and SLM samples.

The measured micro-hardness of the Ti6Al4V alloy was higher for the SLM samples (396 HV) compared to the milled ones (347 HV). The values of the modulus of elasticity of Ti6Al4V alloy, determined by a newly developed methodology consisting of a bending experiment and FEA, are 180 GPa for the milled and 120 GPa for the SLM fabricated samples. They are higher than the data given in the literature or by the manufacturers. Subsequent heat treatment with porcelain firing modes slightly decreased the micro-hardness to 322 HV for the milled and 388 HV for the SLM produced samples and had almost no effect on the modulus of elasticity for both groups.

ACKNOWLEDGEMENTS

The authors express their gratitude to: 1) Desislava Vlasakieva and Miroslav Simeonov from 3D MEDICAL PRINT (Pleven, Bulgaria), who produced the samples by SLM and provided them free of charge for the purposes of this study; 2) Plamen Atanasov and the team of dental laboratory "SMTL-PlamenAtanasov EOOD" (Varna, Bulgaria) for the active free assistance in discussing the experiment and processing the samples.

REFERENCES

[1] Al Hussaini I., Al Wazzan K.A. Effect of surface treatment on bond strength of low-fusing porcelain to commercially pure titanium. The Journal of prosthetic dentistry 94(4) (2005) 350-356

[2] Haag P., Nilner K. Bonding between titanium and dental porcelain: A systematic review. Acta Odontologica Scandinavica 68(3) (2010) 154-164

[3] Dikova T. Nano-engineered coatings on titanium implants. Scripta Scientifica Medica 44(2) (2012) 23-25

[4] Zinelis S., Barmpagadaki X., Vergos V., Chakmakchi M., Eliades G. Bond strength and interfacial characterization of eight low fusing porcelains to cpTi. Dental materials 2010 26(3) (2010) 264-273

[5] Kanazawa M., Iwaki M., Minakuchi S., Nomura N. Fabrication of titanium alloy frameworks for complete dentures by selective laser melting. The Journal of prosthetic dentistry 112(6) (2014) 1441-1447

[6] Dikova Ts., Dental Materials Science, Lectures and laboratory classes notes, Part I, MU-Varna, Varna (2013) 128 p.

[7] Antanasova M., Kocjan A., Kovač J., Žužek B., Jevnikar P. Influence of thermo-mechanical cycling on porcelain bonding to cobalt–chromium and titanium dental alloys fabricated by casting, milling, and selective laser melting. Journal of prosthodontic research 62(2) (2018) 184-194

[8] Dikova T., Dzhendov D., Simov M., Katreva-Bozukova I., Angelova S., Pavlova D., Abadzhiev M., Tonchev T. Modern trends in the development of the technologies for production of dental constructions. Journal of IMAB–Annual Proceeding Scientific Papers 21(4) (2015) 974-981

[9] Antanasova M., Kocjan A., Hočevar M., Jevnikar P. Influence of surface airborne-particle abrasion and bonding agent application on porcelain bonding to titanium dental alloys fabricated by milling and by selective laser melting. The Journal of prosthetic dentistry 123(3) (2020) 491-499

[10] Antanasova M., Kocjan A., Abram A., Kovač J., Jevnikar P. Pre-oxidation of selective-laser-melted titanium dental alloy: effects on surface characteristics and porcelain bonding. Journal of Adhesion Science and Technology 35(19) (2021) 2094-2109

[11] Kazantseva N. Main factors affecting the structure and properties of titanium and cobalt alloys manufactured by the 3D printing. 2018 J. Phys.: Conf. Ser. 1115 042008

[12] Kazantseva N., Krachmalev P., Yadroitsev I., Ezhov I., Merkushev A., Davidov D. Comparative analysis of the structure and properties of titanium and cobalt medical alloys manufacturing by 3D printing. RAPDASA 2019 Conference Proceedings, p. 331-342, ISBN 978-0-6398390-0-4

[13] Zhang X.Y., Fang G., Leeflang S., Böttger A.J., Zadpoor A.A., Zhou J. Effect of subtransus heat treatment on the microstructure and mechanical properties of additively manufactured Ti-6Al-4V alloy. Journal of Alloys and Compounds 735 (2018) 1562-1575

[14] Liu Z., Zhao Z., Liu J., Wang Q., Guo Z., Liu Z., Zeng Y., Yang G., Gong S. Effects of solution-aging treatments on microstructure features, mechanical properties and damage behaviors of additive manufactured Ti–6Al–4V alloy. Materials Science and Engineering 800 (2021) 140380

[15] Wu M.W., Chen J.K., Tsai M.K., Wang S.H., Lai P.H. Intensification of preferred orientation in the additive manufactured Ti-6Al-4V alloy after heat treatment. Materials Letters 286 (2021) 129198

[16] Vrancken B., Thijs L., Kruth J.P., Van Humbeeck J. Heat treatment of Ti6Al4V produced by Selective Laser Melting: Microstructure and mechanical properties. Journal of Alloys and Compounds 541 (2012) 177-185

[17] Kusano M., Miyazaki S., Watanabe M., Kishimoto S., Bulgarevich D.S., Ono Y., Yumoto A. Tensile properties prediction by multiple linear regression analysis for selective laser melted and post heat-treated Ti-6Al-4V with microstructural quantification. Materials Science and Engineering 787 (2020) 139549

[18] Ti Milling Discs, Starbond Ti5 Disc, https://scheftner.dental/starbond-ti5-disc-en.html (17.03.2022)

[19] Technical data sheet, CT PowderRange Ti64 F, Carpenter Additive, 2019 CRS Holdings, Inc., 4 p. www.carpenteradditive.com (17.03.2022)

[20] Datasheet, PowderRange Ti64, Carpenter Additive, 2020 CRS Holdings Inc., www.carpenteradditive.com (17.03.2022)

[21] Gagov Y., Dzhendov D., Parushev I., Dikova T., Geometrical characteristics and density of Ti6Al4V alloy fabricated by milling and selective laser melting. XXVII International Scientific and Technical Conference “Foundry 2022”, Pleven, Bulgaria, 1(4) (2022) 40-45

[22] VITA LUMEX AC, Instruction for use, VITA Zahnfabrik H. Rauter GmbH & Co.KG, Bad Sackingen, Germany, 54 p., www.vita-zahnfabrik.com (Accessed 11.07.2022)

[23] Xu W., Brandt M., Sun S., Elambasseril J., Liu Q., Latham K., Xia K., Qian M. Additive manufacturing of strong and ductile Ti–6Al–4V by selective laser melting via in situ martensite decomposition. Acta Materialia 85 (2015) 74-84