Journal of Achievements in Materials and Manufacturing Engineering

Volume 71 • Issue 2 • August 2015

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Ti6Al4V porous elements coated by polymeric surface layer for biomedical applications

L.A. Dobrzański, A.D. Dobrzańska-Danikiewicz*, T.G. Gaweł

[a] Faculty of Mechanical Engineering, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

* Corresponding e-mail address: anna.dobrzanska-danikiewicz@polsl.pl

ABSTRACT

Purpose: The aim of the paper is to characterise titanium alloy Ti6Al4V coated by polymeric surface layer as a material for biomedical applications. The paper presents a Selective Laser Melting (SLM) method of fabrication of elements to be used as implants from Ti6Al4V powder. It was demonstrated that the metallic scaffolds created have strictly defined geometric dimensions of an object and open pores, and the pores are regular and repeat within the whole volume of the biomimetic element.

Design/methodology/approach: The actual manufacturing process is preceded with creating a model of an element in the stl format that allows to present the element surface by means of a net of triangles. Once the shape is defined of a unit cell and its net parameters, i.e. height, depth and width, they are duplicated with appropriate mathematical algorithms as a result of which a strictly defined, densed and complicated structure of pores defined by a designer is created.

Findings: Scanning electron microscopy was applied for showing the structure of pure scaffolds as well as composites made of Ti6Al4V scaffolds coated by polymeric surface layer. Microscope observations were performed using a SEM Zeiss Supra 35 equipped with EDS detectors for chemical composition analysis.

Practical implications: Manufactured metal-polymer composites can be used in regenerative medicine as biomimetic implants.

Originality/value: The characteristics of biomimetic composites, used in medicine as implants of a palate piece loss with strictly designed geometric shape and dimensions of the object and its strictly planed pores.

Keywords: Scaffold; Porous materials; Laser Melting (SLM); Palate implant; CAMD

Reference to this paper should be given in the following way:

L.A. Dobrzański, A.D. Dobrzańska-Danikiewicz, T.G. Gaweł, Ti6Al4V porous elements coated by polymeric surface layer for biomedical applications, Journal of Achievements in Materials and Manufacturing Engineering 71/2 (2015) 53-59.

MATERIALS

1. Introduction

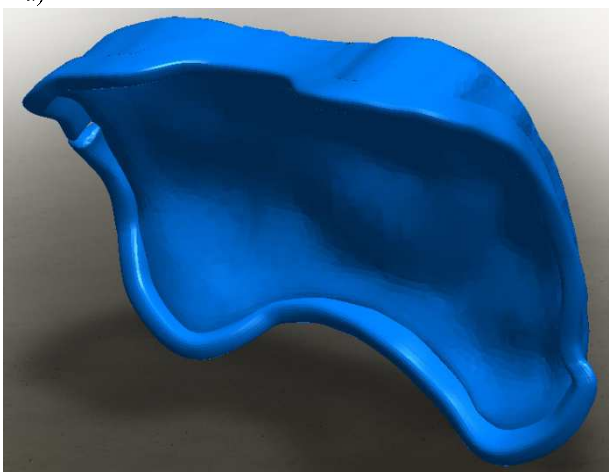
Over 50 years of the history of material engineering and materials science, a paradigm has been formulated providing that, in order to satisfy products' usable functions, it is necessary to design and apply engineering materials which, when undergoing the relevant technological processes of shapening their geometric form and especially structure, ensure the adequate physiochemical properties of the material. The concept of developing, manufacturing, investigating and describing newly developed, biomimetic, laser-sintered engineering materials, is addressing such expectations. Biomedical engineering is a field of science constantly seeking new materials and solutions allowing to improve the existing ones or to create completely new materials with new applications, which will rescue or improve the life of people or animals. At current, one of the trends intensively developed in this field of science are scaffold fabrication methods. Scaffolds are biomimetic materials whose task is to replace and mimic the biological functions of a bone/tissue. Such materials also have a structure of the material being replaced (e.g. open bone pores with a specific structure and geometrical dimensions) and allow the growth of living cells on their surface. The possibility of cells growth is influenced by the porous structure of a scaffold. It allows to supply nutrients to the cells developing on its surface. Scaffolds may be used in an organism in long-term (biocompatible materials) or may be subject to degradation and resorption [1-8].

No implants currently exist which would improve the quality of life of persons with partial palate recesses caused by mechanical injuries, tumorous diseases or cleft palate. Children can be treated surgically, involving an operational procedure. Adults do not have such an alternative. Prostheses made of plastics and/or metals biocompatible with a human organism are currently used. Such prostheses are not durable, uncomfortable and also look unsightly. For this reason, the authors of the article have decided to create a porous implant as a scaffold with a specific shape of open pores. The article presents a method of Computer Aided Materials Design (CAMD) and of the manufacturing of an innovative palate implant made of Ti6Al4V powder for individual needs of a patient with a palate recess by Selective Laser Melting (SLM). A unit cell with predefined shape and geometrical dimensions was modelled during scaffold designing using CAMD software, which enabled to create a model with a strictly designed porous and regular structure having open pores within its entire volume with specific dimensions and geometric shape. The shape and geometrical dimensions of the scaffold produced correspond to a piece of the palate recess of the patient for whom a porous implant was designed individually [1-4,9-12]. A thin layer of PMMA polymer corresponding to the patient's soft issues was deposited onto the lower part of the porous implant in the last stage of works.

2. Computer Aided Materials Design

A process of Computer Aided Materials Design (CAMD) was preceded by a clinical stage whose purpose was to obtain plaster casts from the patient with a palate recess.

a)



b)

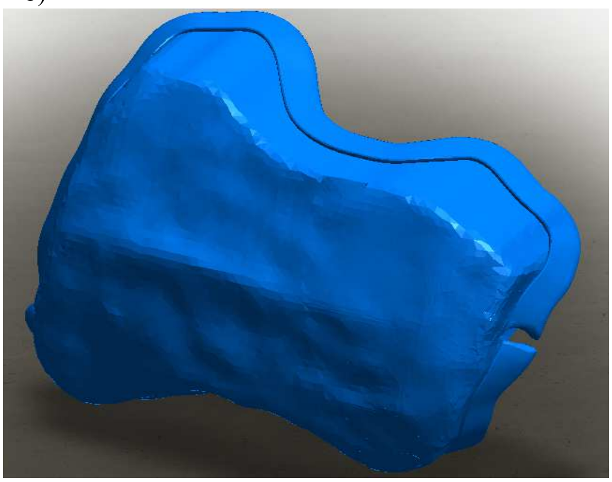


Fig. 1. View of 3D virtual model of solid palate implant; a) bottom view, b) top view

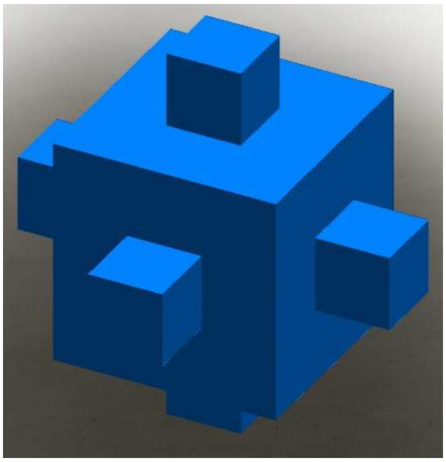
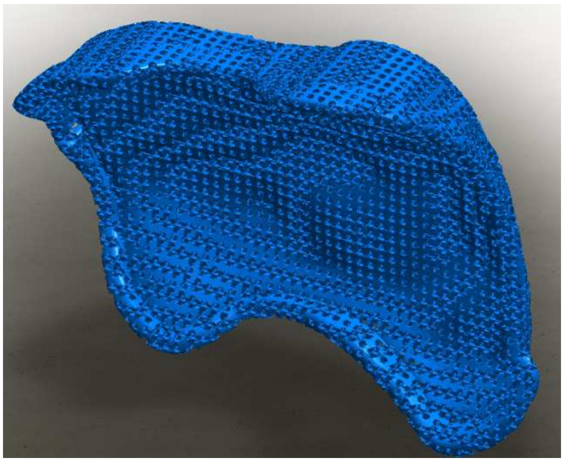


Fig. 2. Newly designed cube-shaped unit cell with six small symmetrically contiguous cubes

a)



b)

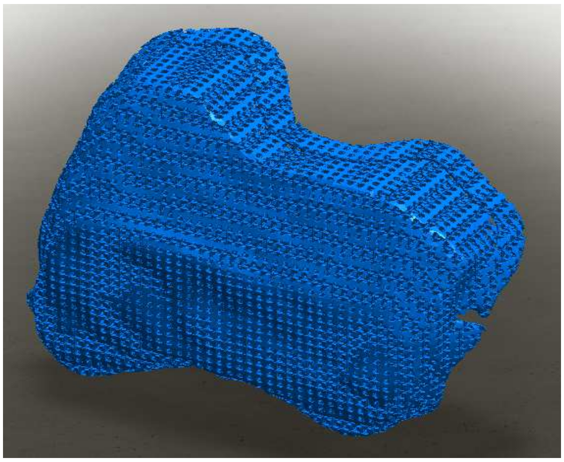


Fig. 3. 3D view of porous scaffold consisting of newly designed unit cells; a) bottom view, b) top view

The produced plaster cast was scanned at the clinical stage to produce its virtual 3D model. Specialist software enabled to design a 3D model of the solid implant whose geometrical dimensions corresponded to the given part of the palate recess. The so established model is shown in Fig. 1. The article authors created a unit cell with a specific shape and geometrical dimensions using CAMD software (Fig. 2).

A unit cell is determining the geometrical dimensions and shape of the scaffold pores. It is important that the cell is of symmetric shape. A unit cell with its arms 100 x 100 µm long and wide, inscribed into a cube with its side 500 µm long, was its arms 100 x 100 µm long and wide, inscribed into a cube with its side 500 µm long, was created with Autodesk Inventor software. The cell core is dimensioned 300x300µm. AutoFab software allowed to convert the virtual model of a solid implant to a porous model according to the unit cell designed in prior. Fig. 3 shows a virtual 3D scaffold model after conversion, i.e. having a porous structure consisting of multiplied unit cells, created with Autofab software.

3. Materials and methodology

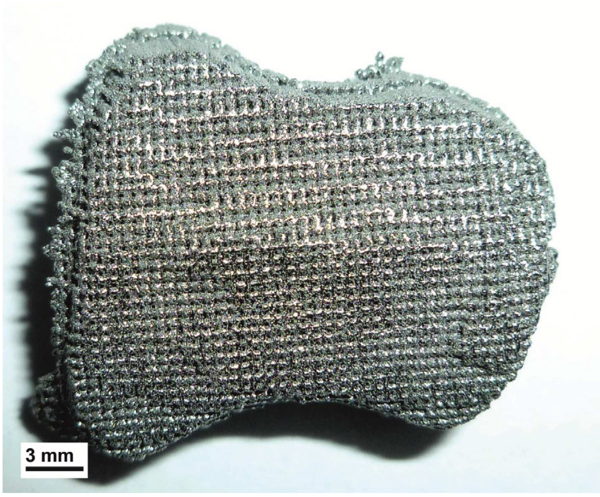
The material used for creating scaffolds by selective laser melting is Ti6Al4V powder with spherically shaped grains whose diameter is between 15 to 45 µm. According to the manufacturer's specification, the titanium alloy used for the experiments is dedicated to medical applications, which is stated by the applicable standard. According to the manufacturer's specifications, the powder used for producing scaffolds by the SLM method contains the following chemical elements: Al (6.35%), V (4.0%), C (0.01%), Fe (0.2%), O (0.15%), N (0.02%), H (0.003%), others (0.4%).

The first stage of works prior to the SLM process is to create a virtual 3D model of a scaffold. The stage is described in chapter 2. Prior to transferring a file with a virtual 3D model of a porous implant to the SLM machine, the model is converted to the net of triangles. This model modification produces inaccuracies and errors. The smaller the field of triangles, the higher accuracy of the part produced, but also the longer production time of the actual object. The so obtained model is still exported to a file with *stl* extension, which can be exported to a device carrying out the selective laser sintering (SLM) process. A file with a relevant extension is transferred to the device via an USB port, and the model is virtually placed in the device's work chamber. This has a large effect on the time and amount of the powder needed to fabricate a part and for supports supporting the model and securing it against collapse. The model is also divided into the number of layers along the model area cross-section relative to the work platform on which it is located. The number of layers is dependent on the thickness of the deposited layer of powder. The key process parameters are determined in the next phase, i.e. laser power, scanning rate, laser point size and thickness of the deposited powder layer. The powder is preheated at 160°C for several hours to remove moisture before commencing the actual SLM process.

4. Results and discussion

A metal scaffold made of Ti6Al4V power is the result of executing the SLM process. The residual powder was removed from the scaffold created using compressed air. Fig 4a presents the created physical object after mechanical removal of supports and after removing it from a work platform.

a)



b)

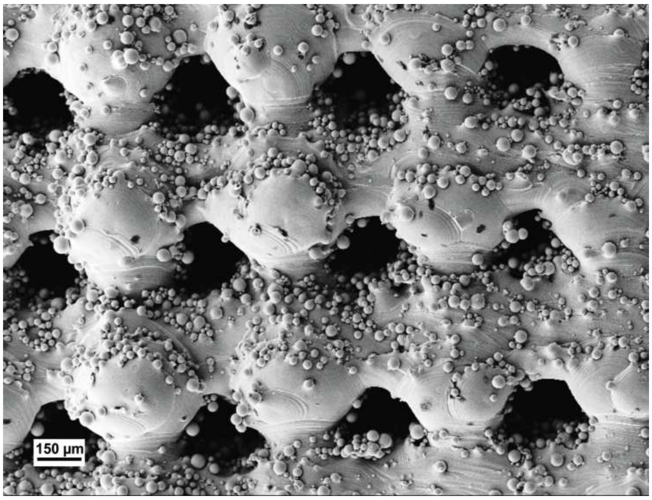
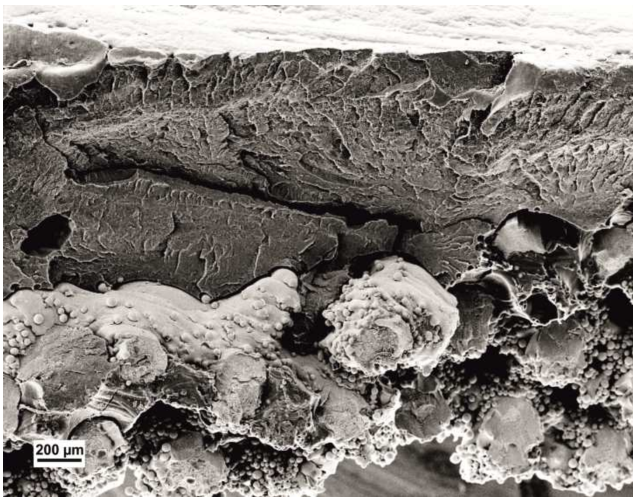
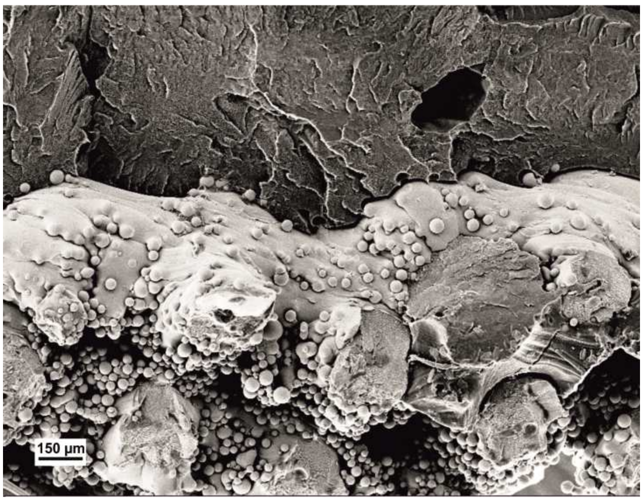


Fig. 4. Metallic scaffold manufactured with SLM method from Ti6Al4V powder; a) macro view; b) Mag. 150x, SEM

a)

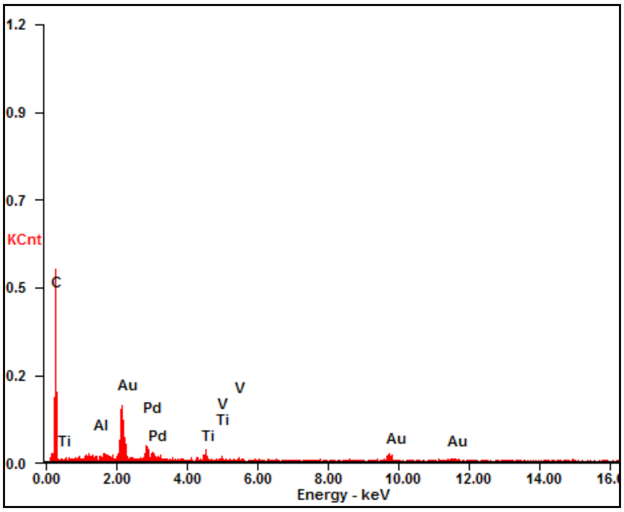


b)

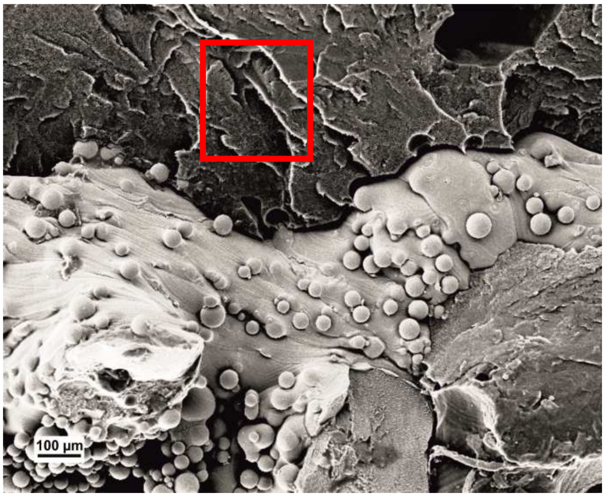


Rys. 5. The newly manufactured metallic-polymer composite; Mag: a) 100x, b) 150x; SEM

a)



b)

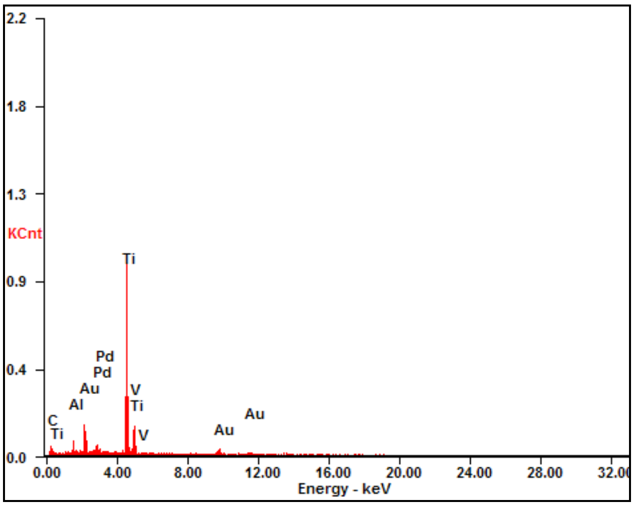


c)

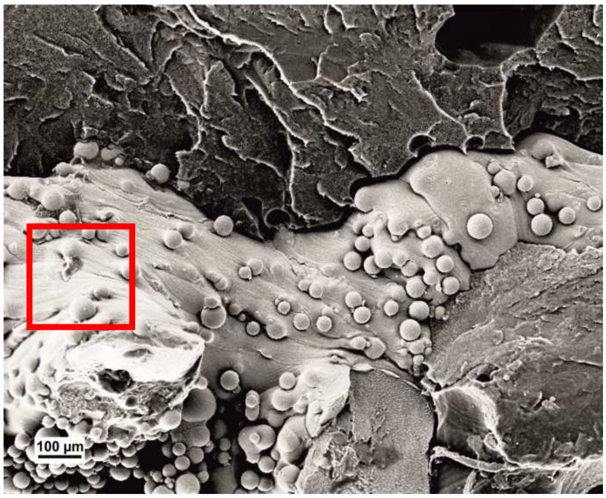
Element	CK	AlK	PdL	TiK	VK	AuL	Matrix
Wt [%]	66.1	00.1	04.6	01.9	00.7	26.7	Correction
At [%]	95.9	00.1	00.7	00.7	00.2	02.4	ZAF

Fig. 6. Qualitative (a) and quantitative (b) chemical composition analysis from the microarea (c), SEM, magnification of 200x made by EDS

a)



b)



c)

Element	CK	AlK	PdL	TiK	VK	AuL	Matrix
Wt [%]	12.2	01.5	03.8	46.4	02.5	33.6	Correction
At [%]	44.4	02.4	01.6	42.2	02.2	07.4	ZAF

Fig. 7. Qualitative (a) and quantitative (b) chemical composition analysis from the microarea (c), SEM, magnification of 200x made by EDS

Surface topography of the scaffolds produced was viewed with a Supra 25 SEM microscope, as shown in Fig. 4b. It was observed that the studied material has a porous, regular latticework-shaped structure. Unmelted grains of powder exist on the surface of the produced scaffolds, which were partly melted into the structure when depositing particular layers of the material in the SLM process. The unmelted powder grains contribute to increased surface roughness and have reduced the size of pores. It was also confirmed that the pores in the scaffolds produced are open.

A thin layer of PMMA polymer was deposited onto the lower part of the implant produced in the last stage of the research works. A microscope image of the specimen fracture, being a newly fabricated metallic-polymer composite, carried out using the scanning electron microscope (SEM), is shown in Fig. 5. The metal part of the composite was gently cut to produce a fracture, and then, after cooling the specimen in liquid nitrogen, it was broken by pressing. The specimen was coated with a layer of gold and palladium to prepare a microscope preparation (SEM). The photographs (Fig. 5) show the permanent bonding of PMMA with a scaffold. An EDS analysis (Fig.6 and 7) has confirmed the presence of C, being a component of the polymer and of Ti, Al and V, being elements forming part of the scaffold. The charts (Fig. 6c and 7c) also show Au and Pd peaks originating from the layers deposited onto the specimen to prepare the microscope preparation.

5. Conclusion

It was indicated in the article presented that modern software installed in the equipment for CAMD and rapid prototyping methods allow to design and fabricate modern implants whose geometrical dimensions are corresponding to recesses in the fragments of individual patients' palates. The user can not only design a shape of a scaffold corresponding to geometrical dimensions of the actual recess in a patient's tissues, but also a shape and size of a unit cell corresponding to scaffold pores. This allows to design a scaffold with a regular structure and specific geometrical dimensions of open pores. A scaffold produced using Ti6Al4V powder has visible powder grains, which have not been fully melted in the SLM process. Works are currently in progress to select the type and concentration of acid which will be used for surface treatment by chemical etching and/or electropolishing to reduce surface roughness of the actual object produced. The authors of this article have also been working to optimise the manufacturing process of an innovative biomimetic composite consisting of a metallic/ceramic scaffold, onto the whole surface of which, or onto parts thereof, a thin polymer layer corresponding to a patient's soft tissues is deposited.

Acknowledgements

The works have been implemented within the framework of the BIOLASIN project entitled "Investigations of structure and properties of newly created porous biomimetic materials fabricated by selective laser sintering" headed by Prof. L.A. Dobrzański, funded by the Polish National Science Centre in the framework of the "Harmony 4" competitions. The project was awarded a subsidy under the decision DEC-2013/08/M/ST8/00818.

Additional information

The research results included in this paper were presented at International Conference on Frontiers in Materials Processing Applications, Research & Technology FiMPART'15, which had place from 12^{th} to 15^{th} June 2015 in Hyderabad, India.

References

[1] International project entitled "Investigations of structure and properties of newly created porous biomimetic materials fabricated by selective laser sintering BIOLASIN" DEC-2013/08/M/ST8/00818.

[2] A. Kaźnica, R. Joachimiak, T. Drewa1, T. Rawo, J. Deszczyński, New trends in tissue engineering, Arthroscopy and Arthritis Surgery 3/3 (2007) 11-16 (in Polish).

[3] R. Nieborak, D. Rolski, E. Mierzwińska-Nastalska, J. Kostrzewska-Janicka, S. Starościak, Prosthetic rehabilitation of patients with soft palate defects following surgery of neoplasms in the maxillofacial region: A case report, Prosthodontic LX/1 (2010) 50-54 (in Polish).

[4] B. Borsuk-Nastaj, M. Młynarski, Selective laser melting (SLM) technique in fixed prosthetic restorations, Prosthodontic LXII/3 (2010) 203-210 (in Polish).

[5] N. Evans, E. Gentelman, J. Polak J, Scaffolds for stem cells, Materials Today 9/12 (2006) 26-33.

[6] S. Padilla, S. Sanchez-Salcedo, M. Vallet-Regi, Bioactive glass as precursor of designed architecture

scaffolds for tissue engineering, Journal of Biomedical Materials Research Part A 81A (2006) 224-232.
[7] M. Schieker, H. Seit, I. Drosse et al., Biomaterials as scaffold for bone tissue engineering, European Journal of Trauma 32 (2006) 114-124.
[8] L.A. Dobrzański, Overview and general ideas of the development of constructions, materials, technologies and clinical applications of scaffolds engineering for regenerative medicine, Archives of Materials Science and Engineering 69/2 (2014) 53-80.
[9] L.A. Dobrzański, A.D. Dobrzańska-Danikiewicz, P. Malara, T.G. Gaweł, L.B. Dobrzański, A. Achtelik, Patent application no. P.411689, Polish Patent Office.
[10] LA Dobrzański, A Achtelik-Franczak, M Król, Computer Aided Design in Selective Laser Sintering (SLS)-application in medicine, Journal of Achievements in Materials and Manufacturing Engineering 60/2 (2013) 66-75.
[11] N. Guo, Mn C. Leu, Additive manufacturing: technology, applications and research needs, Frontiers of Mechanical Engineering 8/3 (2013) 215-243.
[12] G. Pyka, A. Burakowski, G. Kerckhofs, M. Moesen, S. V. Bael, J. Schrooten, M. Wevers, Surface Modification of Ti6Al4V Open Porous Structures Produced by Additive Manufacturing, Advanced Engineering Materials 14/6 (2012) 363-370.