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# FABRICATION OF SCAFFOLDS FROM Ti6Al4V POWDERS USING THE COMPUTER AIDED LASER METHOD

# WYTWARZANIE SCAFFOLDÓW Z PROSZKÓW Ti6Al4V Z UŻYCIEM KOMPUTEROWO WSPOMAGANEJ METODY LASEROWEJ

The aim of the research, the results of which are presented in the paper, is to fabricate, by Selective Laser Melting (SLM), a metallic scaffold with Ti6Al4V powder based on a virtual model corresponding to the actual loss of a patient's craniofacial bone. A plaster cast was made for a patient with a palate recess, and the cast was then scanned with a 3D scanner to create a virtual 3D model of a palate recess, according to which a 3D model of a solid implant was created using specialist software. The virtual 3D solid implant model was converted into a 3D porous implant model after designing an individual shape of the unit cell conditioning the size and three-dimensional shape of the scaffold pores by multiplication of unit cells. The data concerning a virtual 3D porous implant model was transferred into a selective laser melting (SLM) device and a metallic scaffold was produced from Ti6Al4V powder with this machine, which was subjected to surface treatment by chemical etching. An object with certain initially adopted assumptions, i.e. shape and geometric dimensions, was finally achieved, which perfectly matches the patient bone recesses. The scaffold created was subjected to micro-and spectroscopic examinations.
*Keywords*: biomimetic materials, CAMD, scaffolds, SLM, Ti6Al4V powders, SEM, EDS

Celem badań, których wyniki zaprezentowano w artykule jest wytworzenie, metodą selektywnego topienia laserowego (SLM), scaffoldu metalowego z proszku Ti6Al4V na podstawie wirtualnego modelu odpowiadającego rzeczywistemu ubytkowi kości twarzoczaszki pacjenta. Od pacjenta z ubytkiem podniebienia pobierano wycisk gipsowy, który następnie zeskanowano za pomocą skanera 3D, w celu uzyskania wirtualnego modelu 3D ubytku podniebienia, na podstawie którego z użyciem specjalistycznego oprogramowania utworzono model 3D litego implantu. Po zaprojektowaniu indywidualnego kształtu komórki jednostkowej, determinującej wielkość i trójwymiarowy kształt porów scaffoldu, poprzez multiplikację komórek jednostkowych przekształcono wirtualny model 3D implantu litego w model 3D implantu porowatego. Dane dotyczące wirtualnego modelu 3D implantu porowatego przetransferowano do urządzenia służącego do selektywnego topienia laserowego (SLM) i z użyciem tej maszyny z proszku Ti6Al4V wytworzono metalowy scaffold, który poddano obróbce powierzchniowej poprzez trawienie chemiczne. Finalnie otrzymano obiekt o założonych na wstępie: kształcie i wymiarach geometrycznych, które idealnie odpowiadają ubytkowi kości pacjenta. Wytworzony scaffold poddano badaniom mikroi spektroskopowym.

## 1. Introduction

Materials engineering is a field of science encompassing such disciplines as: engineering materials manufacturing technologies and computer-aided design in materials science and materials engineering. Its constant development necessitates the intensive enhancement and optimisation of the existing technological solutions and a search for modern and innovative opportunities of producing new materials or modifying the existing ones. Continuous endeavours have therefore been made to improve physical properties, especially mechanical properties and/or functional properties of the materials produced to ensure their high quality and the market attractiveness of the products manufacturing with them [1-3].

Scaffolds, in which the authors of this publication are interested, are biomimetic materials whose task is to mimic the biological functions of the object replaced. Such materials are also featuring a structure of the material replaced and enable the growth and adhesion of cells. Scaffolds are produced with biocompatible materials which can be employed for a long time in an organism or may be subject to gradual degradation and resorption. A distinctive characteristic of scaffolds is their structure made of open pores. Its task is to ensure access, for cells, to nutrients and to ensure their growth on its surface. It is important that a small diameter of pores needs to be taken into accounting when designing this type of materials [5-12].

The article presents a method of designing metallic scaffolds with their shape and geometric dimensions corresponding to the bone loss of a particular patient using Computer Aided Materials Design (CAMD) and methods of manufacturing such materials from Ti6Al4V powders using an SLM device. The actual elements fabricated feature a strictly de-

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fined shape and geometric dimensions, and also consist of three-dimensional unit cells, as a consequence of which a material manufactured by the SLM method possesses a regular and repeatable structure within its entire volume.

## 2. Computer aided materials design

A design process was initiated by making a plaster cast of a patient with a palate recess, and then a virtual 3D model of a solid implant was designed with CAMD software, the top view of which is shown in Fig. 1, and a bottom view

Fig. 1. Top view of 3D model of solid palate implant

Fig. 2. Bottom view of 3D model of solid palate implant

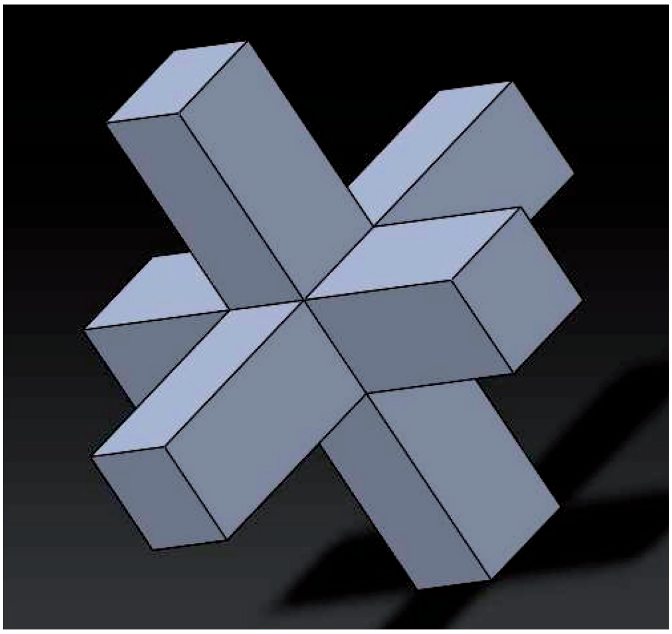


Fig. 3. Newly designed unit cells with the spatial cross shape

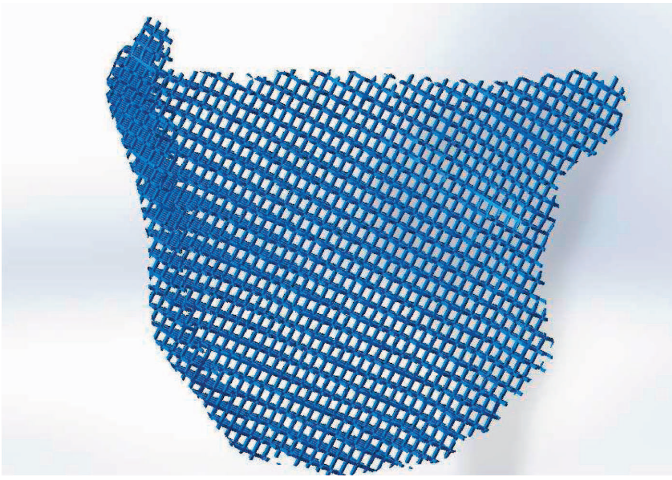


Fig. 4. Top view of 3D model of porous implant consisting of newly designed spatial cross-shaped unit cells

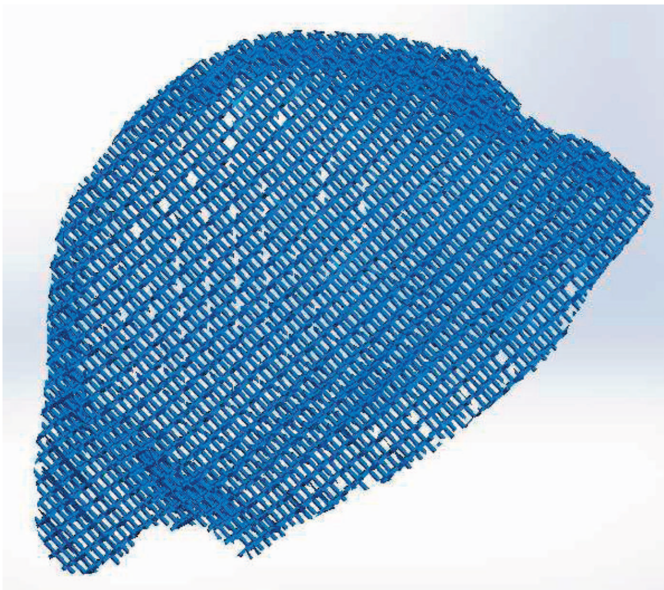


Fig. 5. Bottom view of 3D model of porous implant consisting of newly designed spatial cross-shaped unit cells

in Fig. 2. A literature review [13] points out that unit cells, which are conditioning the shape and size of scaffold pores, have been selected so far using a database bundled with software, e.g. *AutoFab* purchased together with the SLM instrument. The authors of the article have made an attempt to create a new unit cell with its geometric dimensions and shape defined by the designer, which are to ensure appropriate growth of patient cells and their adequate nourishing. A spatial cross-shaped unit cell with its arms 100×100 $\mu$m thick inscribed into a cube with its side 500 $\mu$m long (Fig. 3) was created especially with *SolidWorks*. A model of the newly designed unit cell was recorded in a file with the *stl* extension, and then, with *AutoFab* software; a virtual 3D model of a solid implant was converted into a model of a porous implant with spatial cross-shaped pores corresponding to the modelled unit cell. Fig. 4 (top view) and Fig. 5 (bottom view) show a virtual 3D model of the porous implant with pores having their shape and geometric dimensions corresponding to the designed unit cell.

## 3. Materials and methodology

Ti6Al4V titanium alloy powder is a material utilised for fabricating real objects with an SLM instrument. The chemical composition of the powder according to the manufacturer's specifications is given in Table 1. The powder grains are spherically shaped, and in 93.7% their diameter is within the range of 15÷45 $\mu$m. The powder grain diameter in other 4.3% of cases is larger than 45 $\mu$m, and in the further 2% the diameter is smaller than 15 $\mu$m.

The actual SLM processes was preceded, according to the idea of rapid prototyping, by the creation of a virtual 3D model planned for fabrication of the actual model with CAMD software, as detailed in chapter 2 of this article. The created 3D model is converted into a triangle mesh to transform data from a CAMD system to a SLM instrument, which has adverse consequences such as inaccurate representation of the real object produced. The smaller the triangles the larger the representation accuracy of the designed 3D model, but also as accuracy is rising, so is increasing the fabrication time, and the operator needs to decide about manufacturing process accuracy. Once the virtual model created, converted in prior into a triangle mesh, is exported to the *stl* format, it is virtually suspended in a working chamber of the device in a particular position. Such position is largely influencing the position of the supports supporting the element being produced and secures it against collapse under its own weight and is influencing its manufacturing time, which is the longer the larger is the height of a given object, as measured along the axis $z$. The thickness of the individual deposited powder layer is then identified, and then the model is divided into layers with specific thickness along the cross section of the model in the axis perpendicular to the working chamber substrate in relation to which it had been oriented in advance.

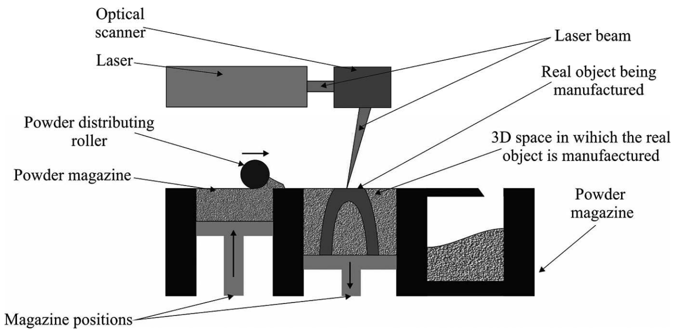


Fig. 6. Schematic of the Selective Laser Melting (SLM) device; an own picture prepared on the basis of [14-16]

Metallic scaffolds manufactured with the SLM method are classified as rapid prototyping methods consisting of selective pre-melting/melting of powders on a point-by-point and layer-bylayer basis [18-19] using a high-performance laser. A schematic of an SLM 250 HL device by SLM Solutions GmbH used for scaffold manufacturing, being the equipment of a research partner of the Silesian University of Technology implementing join research projects [3] – Chemnitz University, Germany, is shown in Fig. 6. Ti6Al4V titanium powder was preheated at 160°C for 12h in the argon protective atmosphere, which ensures that humidity affecting a sintering process is removed. An SLM process was carried out with a scanning rate of 1000 mm/s with the laser power of 100 W using a laser point of 60 $\mu$m in a protective atmosphere of argon.

A process of object manufacturing by SLM was carried out from the bottom. Each layer produced is adhering to the preceding one until the process is completed [17-19]. Powder is fed from a magazine holding loose material, and then distributed at a specific quantity with a shaft travelling across a working platform, which is descending by the exact height of the layer being sintered, whose thickness corresponds to one layer of virtual 3D model section. The excess powder is collected with a roll to a second empty magazine. A computer-controlled laser beam is melting the powder (Fig. 7) in a specifically defined manner and in selectively picked points. A powder layer is deposited and melted selectively in an alternate fashion, until the entire, permanently integrated real object is created. The excess powder, removed from a working platform, can be re-used, by collecting it into a separate special magazine, after being finely sifted over subsequent fabrication processes [17,18].

TABLE 1

Chemical composition of Ti6Al4V powder according to the manufacturer's specification

| **Element** | **Al** | **V** | **C** | **Fe** | **O** | **N** | **H** | **Other aggregately** | **Other each** | **Ti** |
|---|---|---|---|---|---|---|---|---|---|---|
| Concentration [%] | 6.35 | 4.0 | 0.01 | 0.2 | 0.15 | 0.02 | 0.003 | $\leqslant 0,4$ | $\leqslant 0,1$ | Rest |

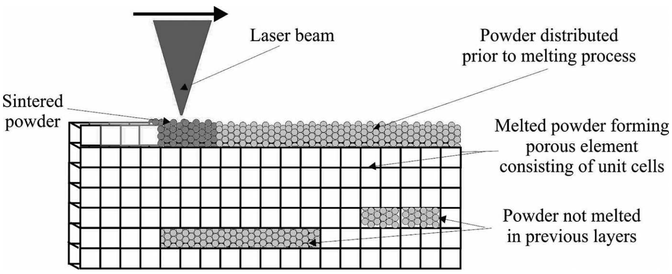



Fig. 7. Sintering of individual powder layers in SLM process

Surface treatment by chemical etching with a hydrofluoric acid (HF) solution was applied to reduce surface roughness ($R_a$) of the newly created scaffold. A solution containing 0.5 ml 70% of HF and 50 g of $H_2O$ was used. The objects manufactured by SLM were placed in polymer containers and subjected to chemical etching for, respectively, 4 and 10 min.

Microscope observations of the titanium alloy powder were carried out with a Keyence VHX-600D light microscope with VH-Z500R lens, and the metallic scaffolds produced by SLM were viewed, prior to and after surface treatment, with a scanning electron microscope (SEM) Supra 35 by Carl Zeiss. The Supra 35 microscope is equipped with an Energy Dispersive Spectroscope (EDS) by EDAX, by means of which a qualitative and quantitative analysis of chemical composition of the newly created porous materials was performed.

## 4. Results and discussion

The single grains of the Ti6Al4V titanium alloy powder which – at the further stage of works – was used for fabrication of metallic scaffolds, were viewed with 1000x magnification using a Keyence VHX-600D light microscope. The results of observations permit to conclude that the particular powder grains are shaped spherically, and their diameter usually ranges over 15÷45 $\mu$m, as shown in Fig. 8.

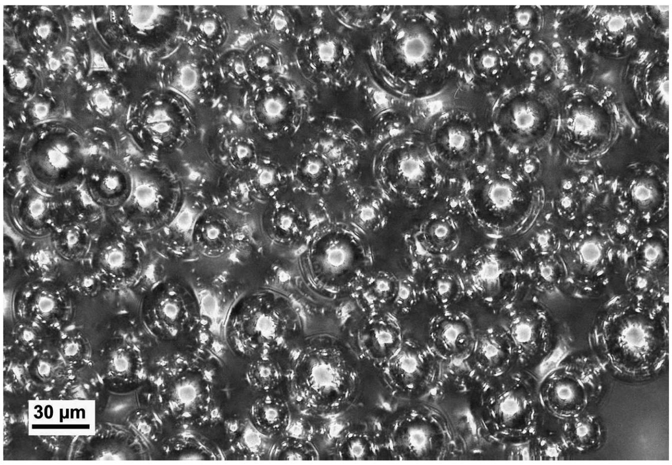



Fig. 8. Ti6Al4V powder; Light microscope; Mag. of 1000x

A metallic scaffold manufactured using Ti6Al4V titanium alloy powder removed mechanically from a working platform and deprived of supports is illustrated in Fig. 9, with top (Fig. 9a) and bottom (Fig. 9b) views.

Surface topography of the scaffolds produced was viewed with a Supra 35 SEM microscope. It was found that the studied material has a porous, regular latticework-shaped structure. It was also found that the pores of the scaffold produced are open (Fig. 10), which was one of the designers' key assumptions. Microscope observations of the studied material's surface topography indicate the presence of singular spherically-shaped powder grains on its surface, which were found there due to adhering to the scaffold surface remelted in the SLM process.

a) b)

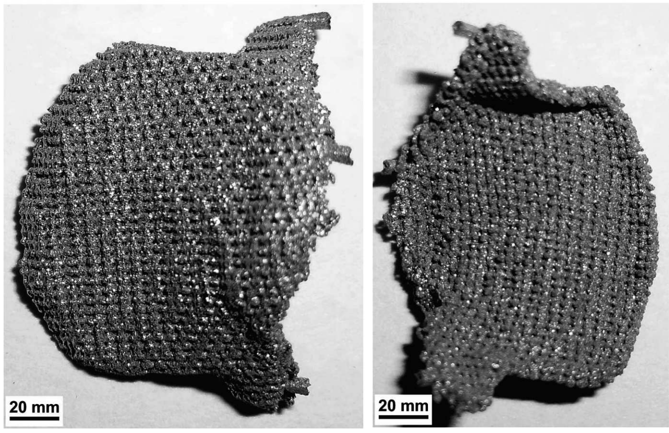



Fig. 9. Metallic scaffold manufactured with SLM method from Ti6Al4V powder; a) top view; b) bottom view

The metallic scaffold after surface treatment performed by chemical etching in a hydrofluoric acid solution with water lasting 4 minutes in Fig. 11 is presented. It was observed that surface treatment of the material reduced the number of titanium powder grains on the material surface. The scaffold after surface treatment performed by chemical etching in a hydrofluoric acid solution with water lasting 10 minutes in Fig. 12 is presented. It was found that single titanium powder grains were completely removed from the surface of the scaffold, and small but visible pits were revealed in the scaffold structure. It was observed that, during chemical etching lasting 10 minutes, single grains of the unmolten powder are etched faster as compared to the actual scaffold structure, which causes small hollows on the material surface in the place where spheroid powder grains existed

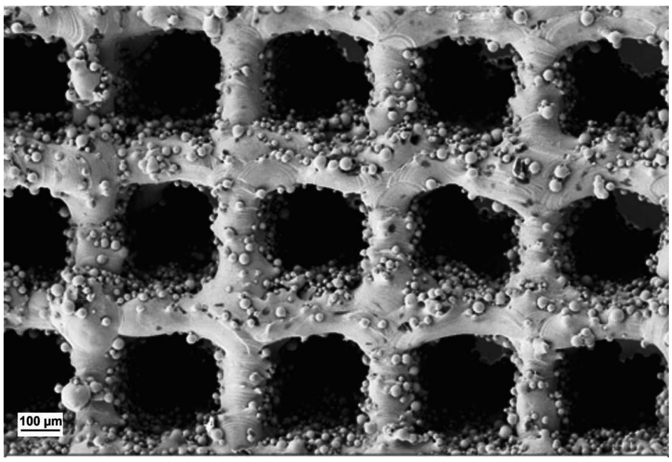



Fig. 10. Scaffold produced with Ti6Al4V powder in SLM, SEM process, 150x magnification

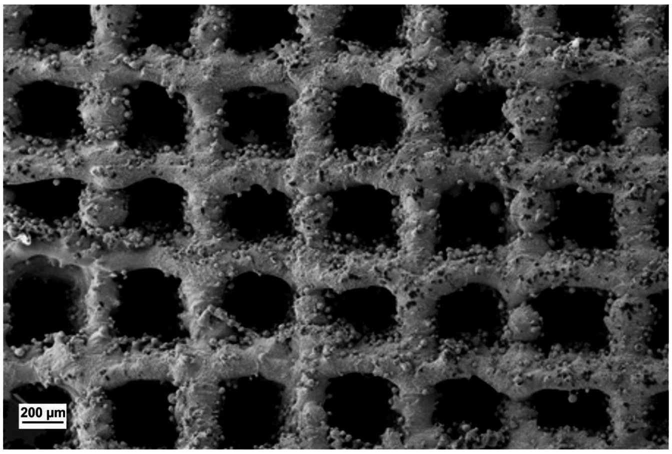



Fig. 11. Scaffold produced with Ti6Al4V powder in SLM process subjected to chemical etching lasting 4 minutes, SEM, 100x magnification

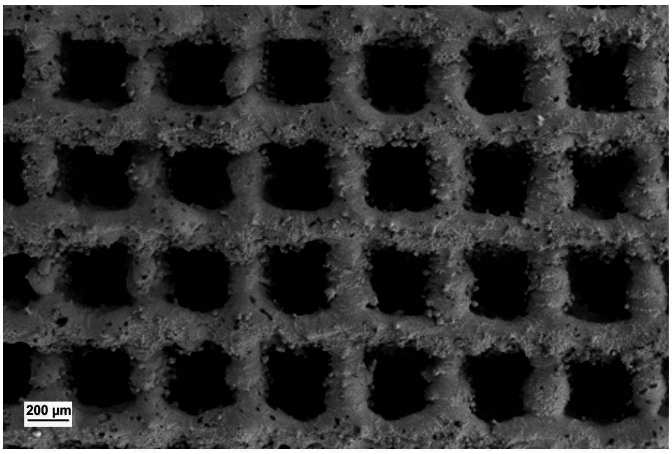
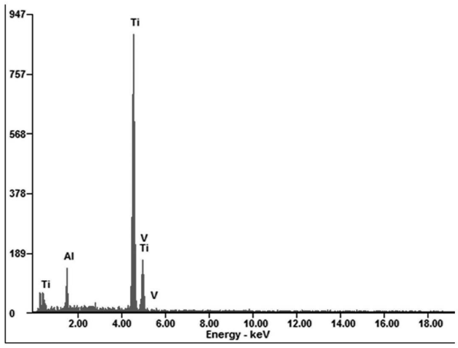



Fig. 12. Scaffold produced with Ti6Al4V powder in SLM process subjected to chemical etching lasting 10 minutes, SEM, 100x magnification

earlier. In order to determine the chemical etching time of the newly produced metallic scaffolds, a series of further experiments has to be undertaken in this scope, optionally combined with another type of surface treatment, i.e. electropolishing.

An Energy Dispersive Spectrometer (EDS) fitted with a SEM Supra 35 microscope was employed for a qualitative and quantitative analysis of chemical composition. The outcomes of the qualitative and quantitative analysis of chemical composition made for two microareas marked on the images made with 100x and 1500x magnification, are presented, respectively, in Figures 13 and 14. The outcomes of the qualitative and quantitative analysis from the two microareas differ negligibly. The qualitative and quantitative analysis results point out in both cases the presence of Ti, Al and V, i.e. elements stated by a powder manufacturer in the technical and commercial specifications. A quantitative analysis indicates that the percentage fraction of elements by mass is approx. 90% of Ti, approx. of 6% and approx. 4% of V, which fully corresponds to the composition of Ti6Al4V powder given by its manufacturer. The chemical composition of the studied material determined in atoms is, respectively: approx. 86% of Ti, approx. 10% of Al and approx. 4% of V.

a)




b)

| Element | AlK | TiK | VK | Matrix |
|---|---|---|---|---|
| Wt [%] | 05.71 | 89.80 | 04.49 | Correction |
| At [%] | 09.73 | 86.22 | 04.05 | ZAF |

c)

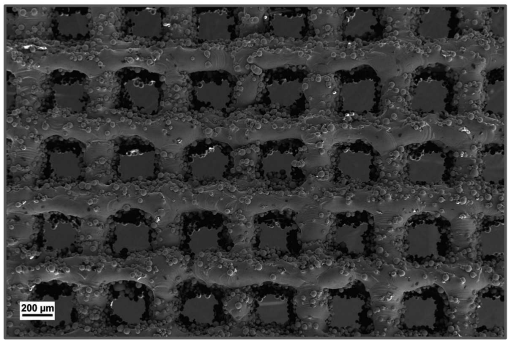
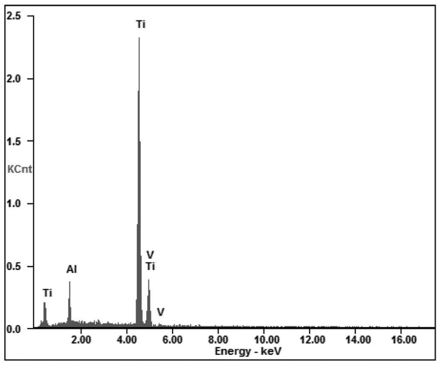



Fig. 13. Qualitative (a) and quantitative (b) chemical composition analysis from the microarea, SEM, 100x magnification (c) made by EDS

a)




b)

| Element | AlK | TiK | VK | Matrix |
|---|---|---|---|---|
| Wt [%] | 06.95 | 89.18 | 03.87 | Correction |
| At [%] | 11.74 | 84.80 | 03.46 | ZAF |

c)

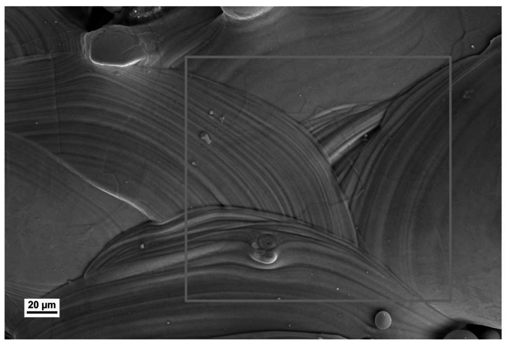



Fig. 14. Qualitative (a) and quantitative (b) chemical composition analysis from the microarea, SEM, 1500x magnification (c) made by EDS

## 5. Conclusions

Modern Computer Aided Materials Design (CAMD) systems allow to design, on a Maketo-Order (MTO) basis, craniofacial implants perfectly matching the loss of bone tissues of a particular patient. A virtual 3D solid implant model can be converted, with special software, into a virtual 3D porous implant model, and a designer can individually design the shape and geometric properties of scaffold pores by creating any unit cell, as exemplified in this article. After converting a virtual 3D model into a mesh of triangles, it is possible to transfer data from a CAMD system to an SLM device and to fabricate a real object whose shape and dimensions ideally match the designed virtual model. Biocompatible Ti6Al4V titanium alloy powder with the grain size of 15÷45 $\mu$m was used for scaffold production and the powder is dedicated to medical applications, as declared by the manufacturer. SEM observations reaffirm that the SLM method allows to manufacture scaffolds with open, regular and recurrent pores possessing defined geometric properties and shape. Surface treatment of a material performed by chemical etching in a hydrofluoric acid solution with water enhances surface quality by eliminating singular spherically-shaped grains found there as a result of adhesion to the scaffold surface remelted in the SLM process. The authors of the article are planning further experiments for surface treatment of scaffolds surface where etching time and acid concentration is adjusted, and where electropolishing is optionally applied.

Intensive works have also been conducted in this thematic field to develop biomimetic composites consisting of metallic/ceramic scaffolds onto the surface of which, or its particular parts, a surface layer being a polymer is deposited, which is a subject of a patent claim of the Authors of this article [4].

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