Research of the properties of highly efficient titanium porous materials using information technologies

Oleksandr Povstyanoy[0000-0002-1416-225X], Nataliya Imbirovich [0000-0001-8276-6349], Rostyslav Redko[0000-0001-5066-0224], Olha Redko[0000-0002-3305-6022], Pavlo Savaryn[0000-0001-7140-7068]

Lutsk National Technical University, 75, Lvivska str., Lusk, 43018, Ukraine
povstjanoj@ukr.net

Abstract. Titanium is characterized by an interesting combination of such properties as high strength, low density, corrosion resistance and biocompatibility. Although the widespread use of titanium at the industrial level has not yet been achieved due to the high costs of its extraction and production. Therefore, titanium is increasingly used in sectors with high demands, such as the aerospace industry or the production of biomedical devices, where the final high cost is not a major factor.

It is believed that the processing of titanium and its alloys using powder metallurgy (PM) methods is a significant way to reduce the cost of manufacturing titanium products, and also provides the opportunity to develop new alloys that are difficult to obtain using traditional technologies.

This work is devoted to the processing of titanium powder from biomedical production waste using various PM methods and is aimed at researching the processing of almost pure, chemically homogeneous and fine-grained titanium-based components.

In particular, the main properties that can be achieved (porosity, microstructure and mechanical properties) and the creation of highly efficient porous materials by advanced methods of isostatic pressing are presented.

Keywords: Titanium, Porous materials, Isostatic pressing, Self-propagating High-temperature Synthesis, Hardness, Microstructure.

1 Introduction

Titanium is a relatively new engineering material compared to other structural metals such as steel and aluminum, as its industrial use only began in the last century. It is an extraordinary metallic element that has many defining characteristics, such as a high melting point (1675°C), relatively low density (4.5 g/cm3), high strength, and good fracture resistance.

Titanium and its alloys are very important industrial metal, they are widely used in aerospace, energy, nuclear industry, food industry, chemical and biomedical engineering, etc. [1]. However, titanium porous permeable materials (PPM) consists of titanium metal and pores, which is why it not only inherits the inherent characteristics of metal, but also provides a number of operational characteristics (filtering, sound absorption, thermal insulation, etc.) [2].

Therefore, titanium PPM is used in the chemical industry due to its resistance to corrosion and filtering properties [3, 4].

In recent years, many scientists have conducted many studies using titanium powders as raw materials for obtaining various types of products and ways of processing them using various methods of powder metallurgy, such as: pressing and sintering [5], isostatic pressing [6] and uniaxial hot pressing [7].
However, crushed titanium feedstock can also be used as a feedstock in powder metallurgy processes, taking advantage of the fact that it is already in powder form. Titanium powders obtained from biomedical production waste are characterized by the presence of chlorides, which create porosity filled with gas during sintering, which prevents the production of completely dense or non-porous titanium products.
The main advantage of using PM methods is the fact that they are end-to-end processes with a higher yield of material and almost without additional mechanical processing, which reduces the costs of manufacturing titanium porous products.

2 Literature Review

Today, there are many ways to obtain porous permeable materials (PPM) from titanium. These are the method of sintering without pressure [8], technology of pressing in dies [9], technology of spatial pressing [10], additive technologies of creating PPM [11]. Among the modern methods of powder metallurgy, the simplest and most common is the method of sintering PPM containing a pore former [12]. Porosity, pore shape, pore size distribution can be adjusted using the concentration of the pore former.
In order to increase competitiveness, expand the scope of application and assortment of titanium PPMs at the current stage of development of the world industry, it is proposed to modify and simplify the processes of obtaining PPMs, in particular during sintering.
The economic efficiency of such PPM from titanium powders is ensured not only due to operational qualities, but also at the stage of their production due to the use of cheap raw materials and energy saving at all stages of the technological process [13].
To determine the cost of titanium porous powder materials, the analysis of costs, which are inextricably linked to the technology, is of great importance.
Thus, the largest share in the cost of production of PPM by powder metallurgy methods is occupied by the costs of basic materials and electricity (80-90%). Traditional methods of sintering titanium PPM require sufficiently powerful furnace equipment with protective media. Sintering costs make up 40-50% of the production cost [14].
These circumstances became a prerequisite for the development of new methods of obtaining highly efficient porous permeable materials from titanium powders obtained from biomedical industry waste **.**

3 Researches Methodology

The raw material was selected for the study - MEDGAL® biomedical engineering waste (**Fig. 1.**). This is pure titanium (99%), waste after processing titanium orthopedic prostheses.

Fig.1. Titanium waste of biomedical engineering MEDGAL®

The technological scheme of biomedical industry waste processing consisted of two stages - grinding and restorative annealing [15].
Grinding was carried out in a vibrating drum mill with an offset axis of rotation. Grinding time was 30, 60, 120 and 180 minutes. The loading mass of the titanium material was determined from the ratio of the mass of the balls to the mass of the powder and was 0.75:1. The degree of grinding was estimated by the amount of titanium powder of one or another fraction.
Restorative annealing to relieve internal stresses in titanium powder lasted 2-2.5 hours at a temperature of 500°C in a vacuum.

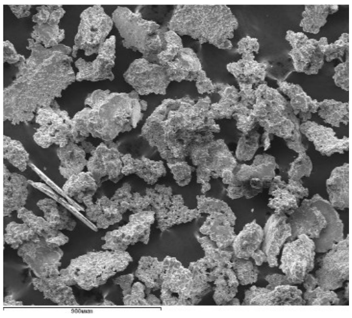

Fig.2. Particles of titanium powder obtained by technology [15].

In the manufacture of titanium PPM that meet modern requirements, the radial pressing scheme is the most rational, and it can be the basis for creating new and improving existing PPM manufacturing technologies, equipment and tools, new types of products.
In order to obtain titanium PPM, an installation for pressing various types of materials was used(**Fig.3.).**

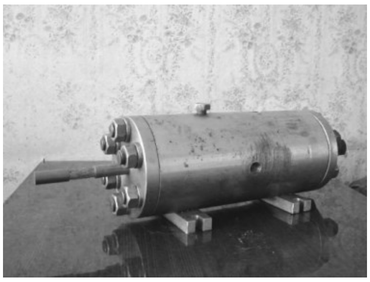

Fig.3. Installation for pressing various kinds of materials

The developed technology for creating porous materials by the method of radial isostatic pressing followed by sintering using self-propagating high-temperature synthesis (SHS) was developed at the Lutsk National Technical University [3, 4]. For the preparation of mixtures, the initial components are titanium powder obtained from biomedical engineering waste (Ti - 98.18%, C - 0.03%, N_2 - 0.08%, H_2 - 0.32%, Si - 0.07%, Ni - 0.14%, Fe - 0.10%) and carbon black (C) – carbon with a bulk density of 0.1 g/cm^3.

The ratio of Ti and C was determined by their atomic weights. The reaction of the formation of TiC by the SHS method from a briquetted mixture of titanium and carbon black due to the high isothermality of the process belongs to the category of combustion reactions that occur in a narrow zone, moving along the briquette due to heat transfer after local initiation of reactions in a heated mixture of reagents. The optimal ratio of Ti and C of the $TiC_{0.5}$ compound was determined experimentally from the point of view of obtaining high-quality products (absence of cracks, warping and obtaining isotropic properties by volume).

The SHS sintering technology of PPM is based on the process of burning powder mixtures (Ti+C) in air without preheating(**Fig.4.**).

Fig.4. Obtaining PPM on the basis of Ti + C with the help of SHS - process

After sintering SHS (t=30 sec) the finished product gradually cools automatically for 30 sec.

On **Fig.5.** presented PPMs from titanium powder – tubes and cones, which were obtained by the method of radial isostatic pressing and SHS-sintering.

Fig.5. General view of PPM based on titanium carbide

The developed and applied technology for obtaining porous materials based on $TiC_{0.5}$ allows to reduce energy consumption both at the pressing stage (by 2 times) and at the sintering stage (by 1.5 times).

4 Results

The properties of porous materials based on titanium carbide are significantly superior to those of porous materials based on pure titanium, obtained in the SHS-burning mode [16]. Porous materials based on $TiC_{0.5}$ have high chemical resistance.
Fig. 6. illustrates the structure of PPM from titanium. Volumetric porosity due to packing of particles and microporosity of particles can be seen. The last circumstance allows to increase the specific surface of the porous material (1 g of porous material - 5 m^2) and, as a result, its sorption properties, which is very important for PPM, which can be used for cleaning liquids and gases.

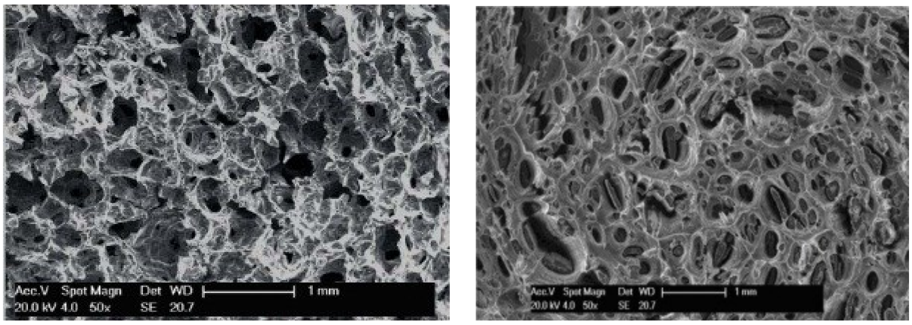

Fig.6. SEM image of the microstructure of titanium PPMs

The microstructure of the obtained material **(Fig. 6)** allows us to state that during the metal reduction reaction it is possible to obtain a porous material with a uniform structure.
One of the most powerful and progressive methods for visualization, obtaining 3D image data of the PPM structure is direct volume rendering, the parameters of light emission and absorption, which are assigned to each point of the image [17]. Modeling the transmission of light through a certain volume of the image allows you to display any data without building intermediate polygonal models.
The creation of a 3D image environment is carried out by superimposing flat cross-sections of the appropriate height range of the finished porous powder material (**Fig.7).**

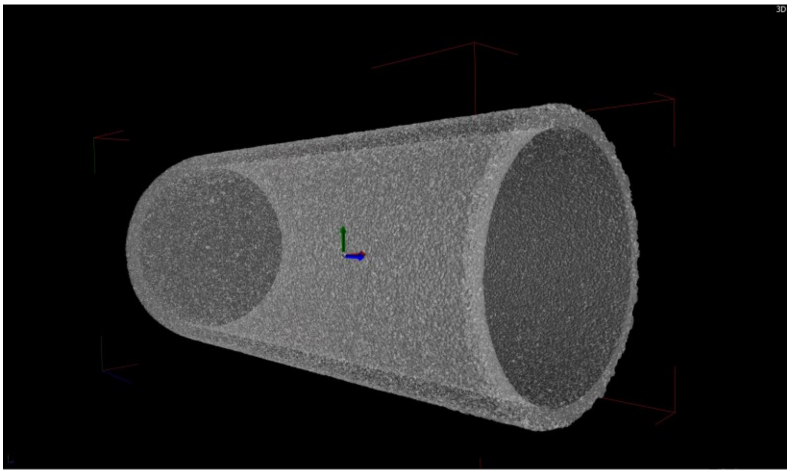

Fig.7. Visualization of the received image of the cross section of the titanium PPM

Metallographic images of PPM grindings are represented by a combination of various structural components. The combination of these structural components (planar and spatial) for PPM are presented in **Fig.8.**

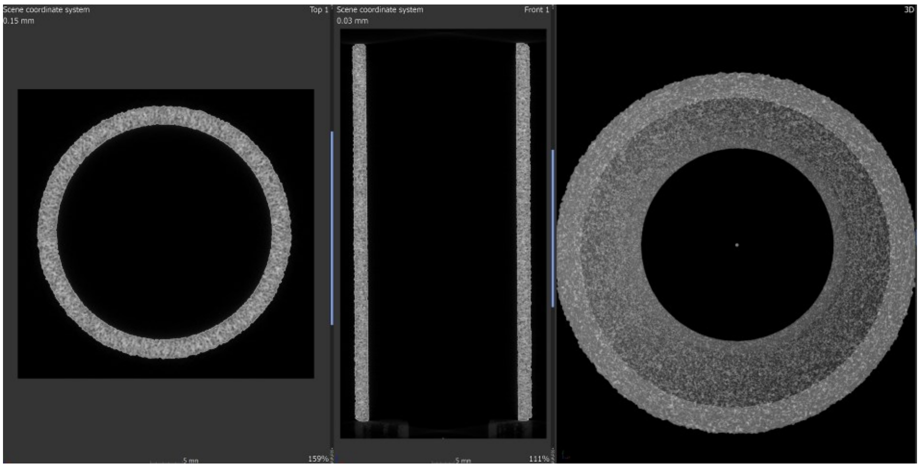

Fig.8. Determination and analysis of structural components of PPM in 3D image format

The morphology of PPMs determines their filtering characteristics and, therefore, their effectiveness in many areas of application. Quantitative and qualitative relationships between the morphology of the porous material, its local and global filtering properties are important in many fields of application [18].
The establishment of quantitative morphological and filtering properties can be based on direct modeling of the porous structure by transfer of the flow of matter in the 3D reconstruction of the material.

The principle of operation is as follows: a section is cut from the PPM sample, generating a clean and smooth surface on the block, which is then displayed on the monitor. After rendering, the new slice is removed from the sample block, giving a new surface to image. This cycle is repeated until the desired number of images is obtained. Typical slice thickness is in the range of 30-100 nm. A total of 1300 slices were obtained, each of which was 100 nm thick and consisted of 4000 × 4000 pixels.
Images were first binarized using an automated sequence consisting of image denoising, filtering, and thresholding [19]. The images were then stitched together to give a representation of the 3D structure of the PPM monolith (**Fig.9**.).
The studied PPM sample consists of a solid metal structure (monolithic skeleton; opaque) with a porosity of ~69% (interstitial voids, shaded).

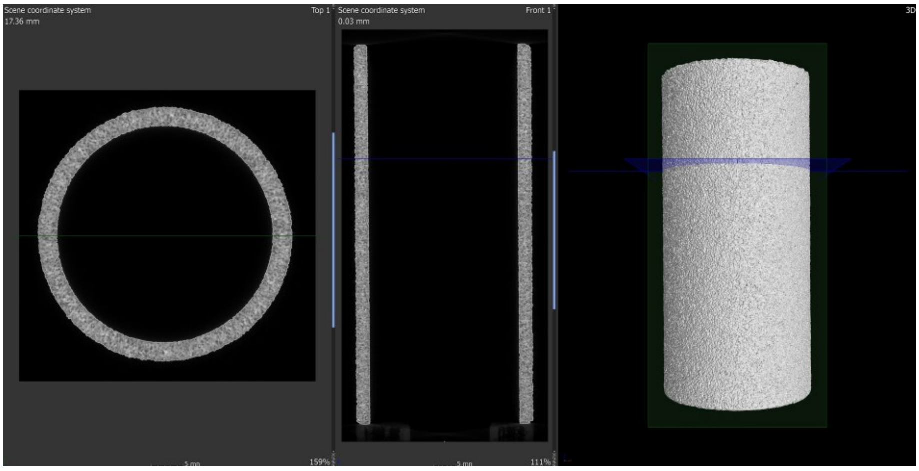

Fig.9. 3D image of the reconstructed volume of the cylindrical sample of PPM

Porosity can be determined from the number of black pixels that represent hollow space divided by the total number of pixels in the PPM structure. The advantage of the image reconstruction approach is its ability to produce spatial tortuous porosity. This makes it possible to study the distributions of radial and axial porosity, namely, perpendicular and parallel to the axis of the cylindrically confined PPM monolith.
Hardness is an important characteristic of a porous material that reflects the bond energy and symmetry of the structure. The hardness of the sintered sample was measured using a Vickers microhardness tester under a load of 1.96 N (300 g) for 15 s. The test results are presented in **Fig. 10.**
The criterion of strength complex is the amount of specific potential energy of shape change accumulated by a deformed object. A dangerous state (flowability) in the general case of a stressed state occurs when the specific potential energy of a change in shape reaches its critical value [20].
With the help of ABAQUS and entering all the necessary data, calculations are automatically carried out and the results of applying pressure from the outside are obtained (**Fig.11.**).

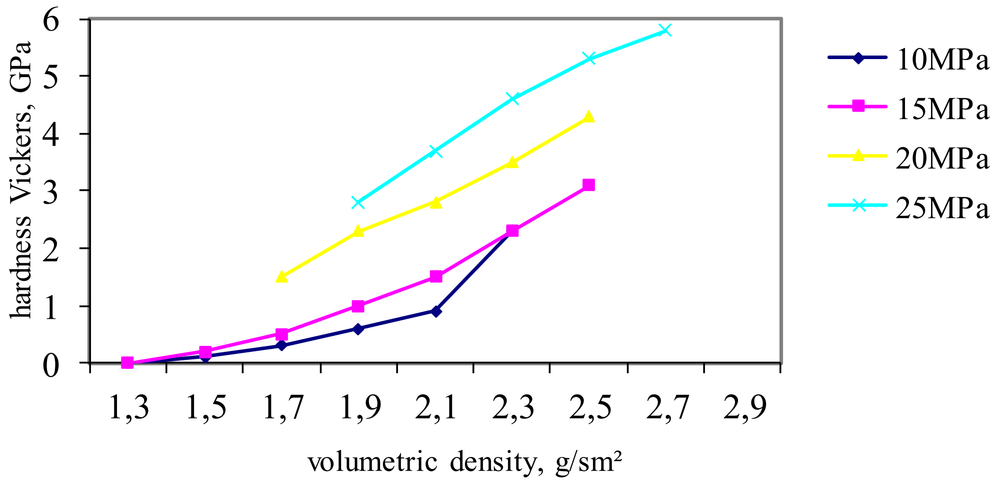

Fig.10. The relationship between Vickers hardness and bulk density

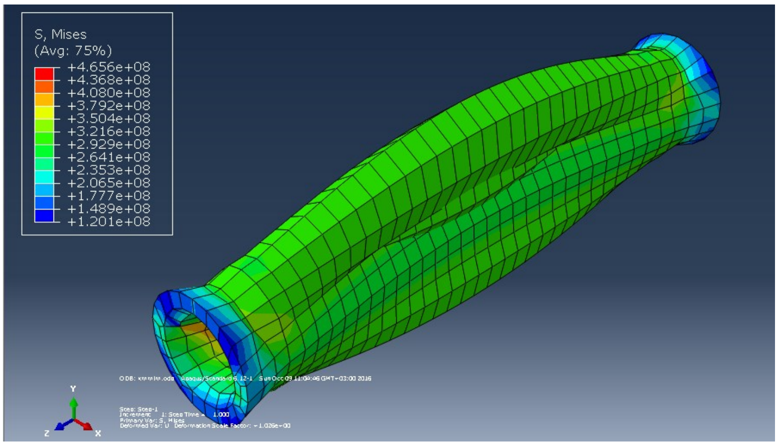

Fig.11. Distribution of load on PPM according to the criterion of maximum stress from the outside

In this case, we get a graphic representation of the distribution of the load on the PPM according to the maximum stress criterion. The detail is displayed in a deformed form. Permissible loads on the PPM according to the maximum stress criterion are shown in green, and exceeding the maximum permissible loads is shown in red.

Let's change the criterion to the criterion of destruction in ABAQUS and display the pressure distribution (**Fig.12**).

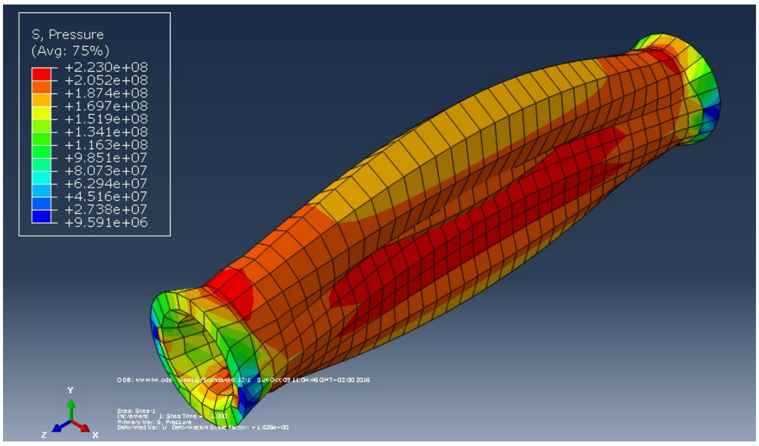

Fig.12. Distribution of deformations along the PPM under the action of a given pressure

The modeling environment provides an opportunity to investigate and predict patterns of structure formation and properties of porous materials, taking into account the sizes of structural elements, establishing correlations between structural components.

5 Conclusions

1. The technology for obtaining highly efficient porous permeable materials based on titanium powders obtained from biomedical engineering waste has been developed. This technology made it possible to reduce energy consumption at the pressing stage by 2 times, at the sintering stage by 1.5 times due to the absence of external energy consumption for sintering (SHS sintering).
2. The formation of a 3D image by superimposing flat cross-sections of the appropriate height range of the finished titanium porous permeable material is shown.
3. A 3D reconstruction and morphological analysis of the PPM was carried out to determine the average value of porosity in the volume and it is 0.687.
4. The main structural characteristics of highly efficient porous permeable materials based on titanium powders were determined - microstructure, porosity, strength.

References

1. Jung, Jae-Hyun.: Study on surface shape control of pure Ti fabricated by electron beam melting using electrolytic polishing. Surface and Coatings Technology 324, 106-110 (2017).
2. Wang, Jianzhong: Energy absorption characteristics and preparation of porous titanium with high porosity. Materials Today Communications 34, 54-60 (2023).
3. Povstyanoy, O., Sychuk, V., Makmyllan, A., Rud, V., Zabolotnyy, O.: Metallographic analysis and processing of images of microstructure of nozzles for sandblasting which are made by powder metallurgy, Powder metallurgy, 3 (4), 234-240 (2015).

4. Rud, V.D., Imbirovych, N.Y., Halchuk T.N., . Chetverzhuk T.I., Smal M.V., Dziubynskyi A.V. Optimization of the Properties of Multilayer Porous Permeable Materials. Mater Sci, 56, 530–535 (2021).
5. ZHANG, Erlin, Xiaoyan WANG, Yong HAN.: Research status of biomedical porous Ti and its alloy in China. Acta Metall Sin 53(12), 1555-1567 (2017).
6. Zabolotny O.: Development of processes of pressing of powder materials. Scientific notes (8*)*, 135-141 (2001).
7. Bolzoni, L., Ruiz-Navas, E.M., Gordo, E.: Processing of Elemental Titanium by Powder Metallurgy Techniques. Materials Science Forum 765, 383-387(2013).
8. Yamanoglu, Ridvan, Abdollah Bahador, and Katsuyoshi Kondoh.: Fabrication methods of porous titanium implants by powder metallurgy. Transactions of the Indian Institute of Metals, 1-13 (2021).
9. Liu, Z., Ji, F., Wang, M., & Zhu, T.: One-dimensional constitutive model for porous titanium alloy at various strain rates and temperatures. Metals, *7*(1), 24-30 (2017).
10. Shbeh, M. M., & Goodall, R.: Open pore titanium foams via metal injection molding of metal powder with a space holder. Metal Powder Report, 71(6), 450-455 (2016).
11. Ataee, A., Li, Y., Fraser, D., Song, G., & Wen, C.: Anisotropic Ti-6Al-4V gyroid scaffolds manufactured by electron beam melting (EBM) for bone implant applications. Materials & Design, 137, 345-354 (2018).
12. Stanev, L., Kolev, M., Drenchev, B., & Drenchev, L.: Open-cell metallic porous materials obtained through space holders—Part II: Structure and properties. A review. Journal of Manufacturing Science and Engineering, 139(5) (2017).
13. McMillan A. J, Archer E, McIlhagger A, Lelong G.: Strength knockdown assessment of porosity in composites: modelling, characterising and specimen manufacture. Journal of Physics: Conference Series. 3. 20-27 2012.
14. Wang, X. S., Lu, Z. L., Jia, L., & Chen, J. X.: Preparation of porous titanium materials by powder sintering process and use of space holder technique. Journal of Iron and Steel Research International, 24(1), 97-102 (2017).
15. Rud' V., Gal'chuk T., Povstyanoi, O.: Powder metallurgy use of waste from bearing production. Powder Metallurgy and Metal Ceramics. 44(1-2), 88–92 (2005).
16. Oh IH, Nomura N, Masahashi N, Hanada S. Mechanical properties of porous titanium compacts prepared by powder sintering. Scripta Materialia 2003; 49: 1197-1202.
17. Korniy, V.: Model and algorithm for processing color metallographic 3D images. Computing. 7(1). 164-170 2008.
18. Xiao, J., & Qiu, G. B.: Research review of space holders of sintered titanium foams with large pores and high porosity. Materials China, 37(5), 372-378 (2018).
19. Bewerse, C., Emery, A. A., Brinson, L. C., & Dunand, D. C.: NiTi porous structure with 3D interconnected microchannels using steel wire spaceholders. Materials Science and Engineering: A (634), 153-160 (2015).
20. Niespodziana, K.: Synthesis and properties of porous nanocomposites Ti-20% HA. J. of Materi Eng and Perform. 28, 2245–2255 (2019).