Porous volumetric structures obtained by additive manufacturing technologies

Kamil Kaszyca[1], Wojciech Danilczuk[2], Rafał Zybała[1,3]

The goal of our work was to develop bulk structures characterized by a variable, controlled porosity, using additive manufacturing techniques (3D printing). A technology for the fabrication of bulk materials with controllable porosity has been developed. For that purpose, the samples with constant porosity were designed and then prepared, which allowed us to learn the possible limit values. Thus, we were able to optimize the design process at the stage of the preparation of the gradient structures.

Key words: 3D printing, porous materials, gradient materials, design of porous structures

Porowate struktury przestrzenne otrzymywane technikami wytwarzania przyrostowego

Niniejsza praca przedstawia proces opracowanie struktur przestrzennych charakteryzujących się zmienną, sterowaną porowatością, z wykorzystaniem technik wytwarzania przyrostowego (druku 3D). W ramach pracy opracowana została technologia wytwarzania materiałów o sterowanej porowatości. W tym celu zaprojektowane i wykonane zostały próbki o stałej porowatości. Pozwoliło to na poznanie możliwych do uzyskania wartości granicznych, co w konsekwencji skutkowało możliwością optymalizacji procesu projektowania na etapie tworzenia struktur gradientowych.

Słowa kluczowe: druk 3D, materiały porowate, materiały gradientowe, projektowanie struktur porowatych

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1. Introduction

Additive manufacturing techniques are at the center of interest for many research groups. For instance, the authors of the recent report [1] mention a prediction that in the years 2016-2020 the market value of 3D printing will increase from 6.1 billion dollars to 21 billion dollars. The article describes the examples of the applications of numerous common techniques, including fused deposition modelling, inkjet printing, stereolithography and selective laser melting/sintering. The possibilities of the use of these techniques for processing of a variety of materials were presented, i.e. polymers (PLA, ABS, resins), metals (Ti-Al-V), ceramic, concrete and composites. Due to the fact that the additive manufacturing techniques can be easily adapted to various purposes and the ability to rapidly obtain customized parts, these methods have a ide range of applications, not only for the production of mechanical elements. The possibility of using electrically conductive polymers enables printing of functional components of the electronic devices, eventually serving as e.g. integrated printed circuit boards [2]. The materials with a variable porosity can be applied as the intermediate products to be used for the production of porous ceramics. They may be intended for metal infiltration or utilized as filters, bone implants [3] or the substrates to be applied in bionics [4, 5]. The composites with a polymer matrix [6] are also used in bionics or in the aircraft industry. The real advantage of the proposed method is that the user has the possibility to obtain a part with a well-defined, customized geometry and structure [7-9], and with high specific strength [10]. Today, a dynamic development of the additive manufacturing techniques is related to a steady increase in their popularity and accessibility. The results of the preliminary studies, presented below, contribute to the growth of the methodology enabling the application of the additive manufacturing techniques for the fabrication of the functionally gradient materials.

The goal of our research was to elaborate a technology to produce the plastics with a controlled open porosity.

[1] *Łukasiewicz Research Network - Institute of Electronic Materials Technology*, 133 Wólczyńska Str., 01-919 Warsaw, Poland e-mail: kaszyca@itme.edu.pl
[2] *Lublin University of Technology, Faculty of Mechanical Engineering, Department of Automation*, Nadbystrzycka 36, 20-618 Lublin, e-mail: wojciech.danilczuk@pollub.edu.pl
[3] *University Research Centre "Functional Materials", Warsaw University of Technology*, Wołoska 141, 02-507 Warsaw, Poland; e-mail: rafal.zybala@pw.edu.pl

At the stage of the design of experiments, the following assumptions were made:

1. The scientific and technological work will enable obtaining a material with an assumed porous structure
2. The control of the process parameters makes it possible to obtain a structure with a gradient porosity
3. A porosity limit value exists that allows open porosity to be obtained.

The article presents the results of the technological and research work carried out in the framework of the project "Porous spatial structures obtained by additive manufacturing techniques", performed at the Institute of Electronic Materials Technology (ITME) as a part of the statutory work. The fabrication of a plastic part with a gradient porosity was considered a criterion for the success of the project. The materials described here were manufactured with a device utilizing the FDM Wanhao Duplicator i4. The material chosen for the tests was Polyactide, made by the Polish company Propox, characterized by a negligible shrinkage during cooling. The batch files for the apparatus were prepared using the IceSl slicer.

2. Methodology for materials fabrication by FDM technique

The materials described in our article have been obtained by the FDM (fused deposition modeling) method, which consists in the formation of a part by a proper deposition of the layers of a plasticized thermoplastic material. After the deposition of each layer, the working platform with the piece being printed moves away from the print head by a distance equal to the thickness of the deposited layer. The devices operating with the use of the FDM technique utilize polymers and their composites as the building materials.

The main parameters responsible for the thickness and density of the filling of a solid are: a factor called INFILL and the layer height (L_h). Additionally, the most important technological parameters are: V - the nozzle linear velocity, T_h - the nozzle temperature and T_b - the substrate temperature. Figure 1 shows the dependence of the planar fill on the INFILL parameter for a single layer (a, b, c) and for two layers (d, e, f).

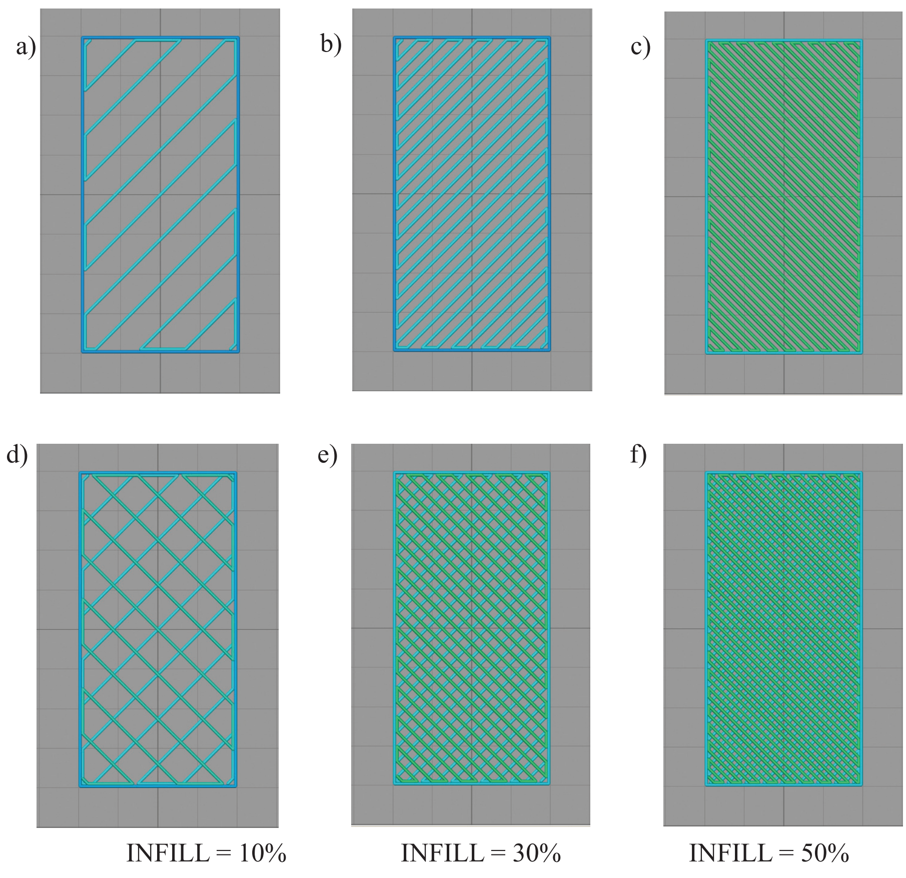


Fig. 1. The planar fill - INFILL factor dependence.
Rys. 1. Zależność wypełnienia od współczynnika INFILL.

As demonstrated on the model example, it is still possible to obtain a structure with open porosity even in the case of 50% filling. The model-based predictions indicated that with the perfect reproduction of the expected layer, any value of INFILL less than 100% would not lead to closed porosity. Due to the discrepancies between the model and the real data, as well as because of the practical aspect of our investigations, later in the report we will focus on the description of the actual conditions and the presented information will be based on the experimental results.

3. Experimental

The goal of the studies reported in this article was the production of a part with a continuously graded porosity. In the described work a series of the samples was made for the following purposes:

1) The examination of the effect of the deposited layer height on the size and the contribution of the pores
(from sample TEST-01 to sample TEST-05)

2) The examination of the effect of the INFILL value on the size and the contribution of the pores
(from sample TEST-01 to sample TEST-05)

3) Preliminary searching for the limit INFILL values that could allow open porosity to be obtained
(from sample TEST-06 to sample TEST-15)

4) The examination of the dependence between the INFILL value and the planar void fraction of the porous zone
(from sample TEST-06 to sample TEST-15)

5) Detailed searching for the limit INFILL values allowing open porosity to be obtained (a series of test samples with discontinuously graded porosity)

Table 1 shows a list of samples prepared during the first stage of the investigations. The tests were performed in order to verify a relationship between the chosen parameters of printing (L_h, INFILL) and the geometry of the pores of the obtained sample, as well as to find the limiting process parameters corresponding to open porosity. The test samples produced at this stage of work had a form of a rectangular prism with the dimensions of 20 × 10 × 10 mm.

Tab. 1. A list of the investigated samples.
Tab. 1. Lista próbek do badań.

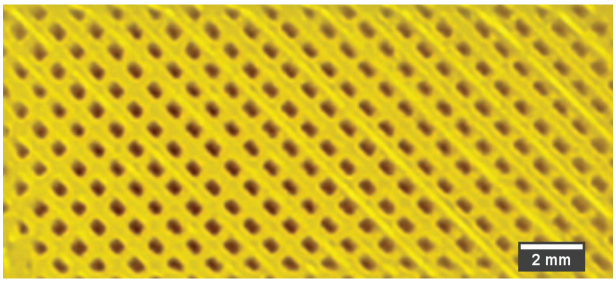
ID	Material	Conditions					
		T_h[°C]	T_b[°C]	L_h[°C]	INFILL	V [m/s]	Substrate
TEST-01	PLA	195	65	0.15	50%	0.037	Glass plate „Float", thickness 4 mm
TEST-02	PLA	195	65	0.2	40%		
TEST-03	PLA	195	65	0.2	50%		
TEST-04	PLA	195	65	0.2	60%		
TEST-05	PLA	195	65	0.25	50%		
TEST-06	PLA	195	65	0.2	10%		
TEST-07	PLA	195	65	0.2	20%		
TEST-08	PLA	195	65	0.2	30%		
TEST-09	PLA	195	65	0.2	40%		
TEST-10	PLA	195	65	0.2	50%		
TEST-11	PLA	195	65	0.2	60%		
TEST-12	PLA	195	65	0.2	70%		
TEST-13	PLA	195	65	0.2	80%		
TEST-14	PLA	195	65	0.2	90%		
TEST-15	PLA	195	65	0.2	100%		

T_h - the nozzle temperature, T_b - the substrate temperature, L_h - the layer height, INFILL - the infill factor, V - the nozzle velocity

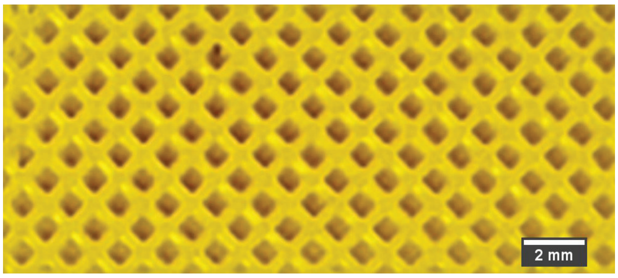
3.1. Pore geometry analysis

Pore geometry analysis was based on a series of test samples TEST-01-TEST-15. The presented structures are oriented in the XY plane of the printer on a glass substrate. At this stage of research, the influence of two parameters on the geometry of pores in the resulting material was examined. The study of pore geometry was based on the analysis of the cross-sectional photographs of the produced materials.

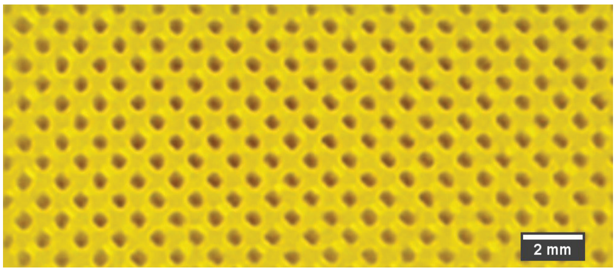
Figure 2 shows the photographs of the analyzed samples listed in Table 1.



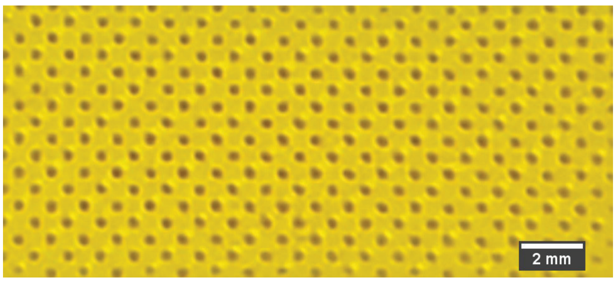
a) TEST-1



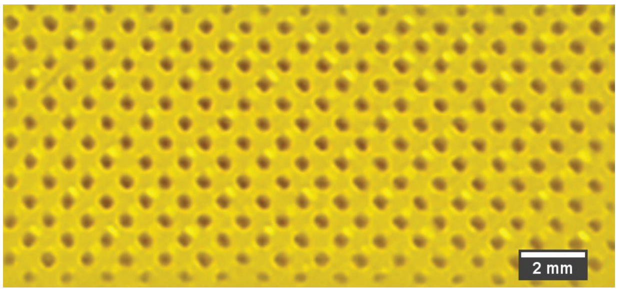
b) TEST-2



c) TEST-3



d) TEST-4



e) TEST-5

Fig. 2. Photographs of the surface of the analyzed samples.
Rys. 2. Fotografie powierzchni analizowanych próbek.

On the basis of the photographs, a binary mask was prepared, where the white region represented the presence of a polymer and a black region indicated the lack of a polymer. The histogram of the mask enabled the determination of the material porosity, while the analysis of the particles provided information on the average pore size ($A_{śr}$). Further steps of an exemplary process of the image analysis are presented in Figure 3. The analysis results are collected in Table 2. For the image analysis an ImageJ software was used.

a)
b)
c)

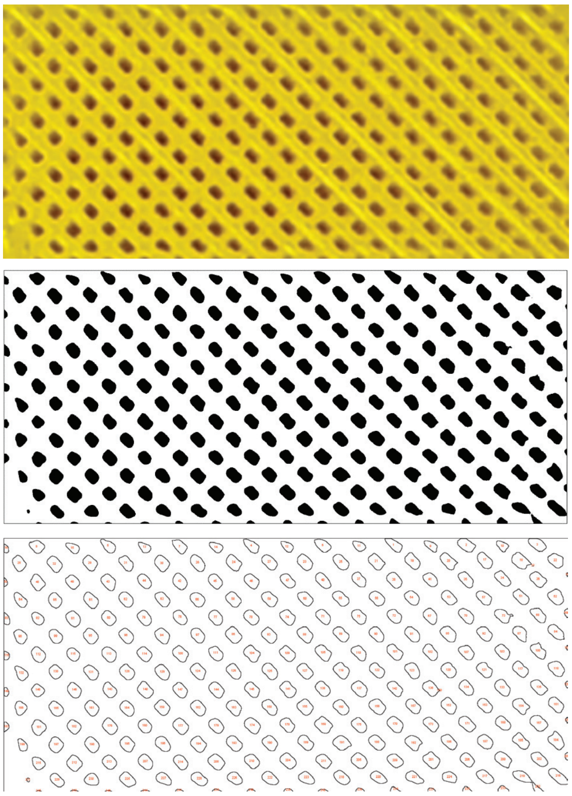


Fig. 3. The stages of PSD analysis.
Rys. 3. Poszczególne etapy analizy obrazu.

Tab. 2. The results of image analysis.
Tab. 2. Wyniki analizy obrazu.

Sample ID	L_{height} [mm]	Infill	Sum [px]	White region [px]	Black region [px]	Number of pores [-]	Average pore size [px]	Average pore size [mm²]	Planar void fraction [%]
TEST-1	0.15	50%	1300328	994661	305667	246	162.69	0.155	23.5
TEST-2	0.2	40%	1255464	865857	389607	159	157.1	0.307	31.0
TEST-3	0.2	50%	1255616	981092	274524	225	157.1	0.153	21.9
TEST-4	0.2	60%	1209632	999178	210454	323	151.3	0.082	17.4
TEST-5	0.25	50%	1298772	1053066	245706	240	162.5	0.128	18.9

In order to examine the relationship between the values of INFILL and the planar void fraction for the material, an image analysis was performed for the series of samples from TEST-6 to TEST-15. The results of the analysis of the structures are presented in Figure 4. It can be seen that the planar void fraction of the porous structure formed depends in a nonlinear manner on the values of INFILL.

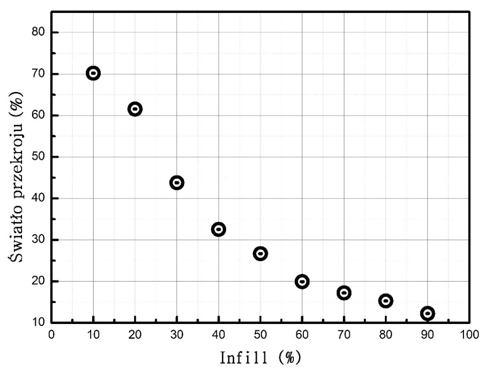


Fig. 4. The planar void fraction - INFILL factor dependence.
Rys. 4. Zależność pomiędzy światłem przekroju a współczynnikiem INFILL.

Analysis of the boundary conditions that allow open porosity to occur

A starting point of our study was the determination of the filling limit value that can ensure the existence of open porosity. For this purpose, a series of test samples with different INFILL values was made. Figure 5 shows an exemplary photograph of a cross-section of a test sample made of 12 zones with the consecutive INFILL values equal to 10%, 15%, ... 70%. The presented structure was obtained by cutting the test sample with a diamond saw in the ZX plane and then grinding the uncovered plane to remove the material exposed to a deformation as a result of cutting.

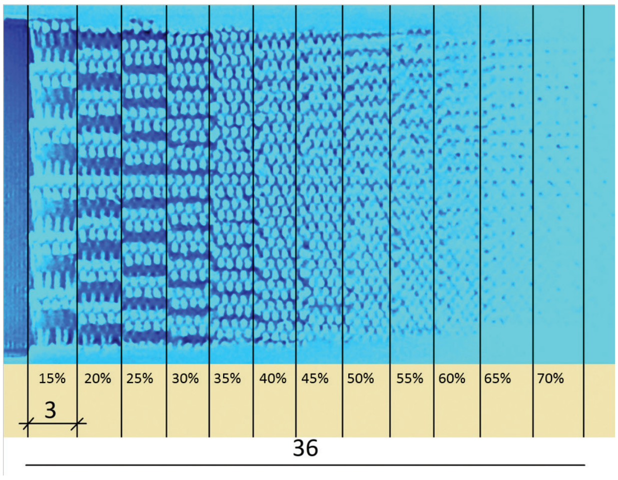


Fig. 5. A photograph of a cross-section of an exemplary test sample.
Rys. 5. Fotografia przekroju przykładowego testera.

To verify permeability of the layers, a series of test samples was prepared with INFILL values ranging from 60% to 90% at the intervals of 5%. It was determined experimentally that the limit value of the layer permeability for gases was 85%. However, a significant increase in the flow resistance was observed already at 70% filling.

Fabrication of an open-graded porous part

On the basis of the data obtained at the stage of the analysis of the samples from the TEST-XX series and the test samples with discontinuously graded porosity, a model of a gradient material has been developed. The elaborated model was a 25 × 50 × 30 mm rectangular prism with a gradient value of INFILL, ranging from 10% to 60%. The values of the planar void fraction of that material varied between 76% and 17%. Figure 6 shows a photograph of a cross-section of the aforementioned gradient material.

The obtained results make it possible to design a structure with a controllable porosity, previously determined at the stage of the porosity design. The desired nature of the porosity changes can be arbitrary (within the technical possibilities of the FDM method).

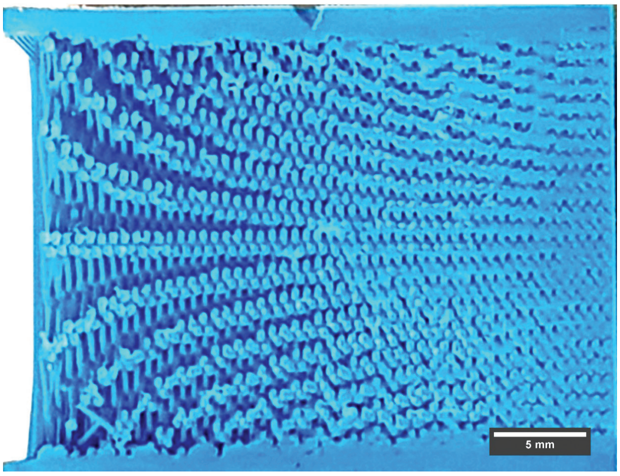


Fig. 6. A photograph of a cross-section of a gradient material.
Rys. 6. Fotografia przekroju otrzymanego detalu o gradientowo zmieniającej się porowatości.

The elaborated technology enables formation of a part with any kind of geometry and with a porosity gradient changing along one of the axes of the part. It is possible to utilize the obtained results for the production of ceramic foams. According to the proposed methodology, a porous structure would be filled with a ceramic material and then the matrix would be removed by its thermal decomposition. A prefabricated product formed in that way, subjected to free sintering process, would be brought to the form of a polycrystalline ceramic material with open porosity. Potentially, the aforementioned products can also find an application as filters. It is possible to use a polymer with silver or titanium oxide particles, which after their activation by UV radiation may purify the flowing fluid from the microorganisms.

4. Conclusions

The results reported here indicate the possibility of utilizing a popular method of additive manufacturing for the production of porous materials. Our article also presents a methodology for optimizing the printing parameters. Figure 7 shows photographs of the exemplary structures.

The use of additive manufacturing techniques to produce gradient materials is a novel idea. Our paper describes the preliminary studies on the applicability of the FDM technique for the fabrication of porous materials. Further work will be focused on the extending of the range of materials by including metals and

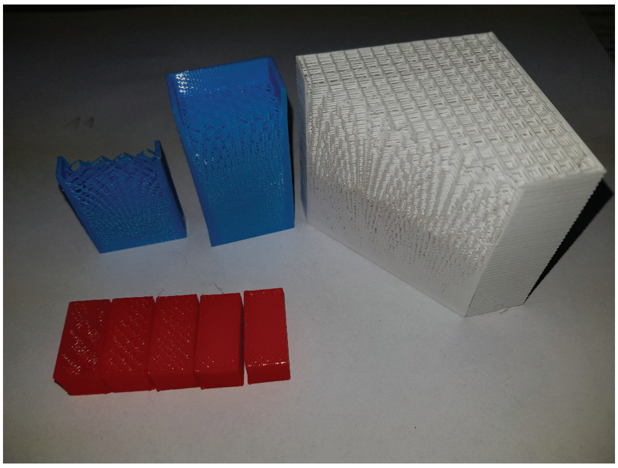


Fig. 7. A photograph of the exemplary structures.
Rys. 7. Fotografia przykładowych struktur.

ceramics, as well as on functionalization of the obtained structures. The analysis of the experimental results has revealed the following relationships:

1. It has been established that the limit value of the INFILL coefficient is 65%, which allowes for a free transport of fluids and powders.
2. With an increase in the layer height L_h at a constant value of INFILL factor, the values of both average pore size and planar void fraction of the structure decrease, whereas the number of pores increases.
3. An increase in the value of the INFILL factor results in an increase in the number of pores and a decrease in both the average pore size and the planar void fraction of the structure.

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