Research of composition porosity based on 3d-printed frames and impregnated with epoxy resin

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**Annotation.** The paper discusses a method of increasing the strength of 3D-printed structures made by the fused deposition modelling process, which consists in vacuum impregnation of printed products in a compound based on epoxy resin. Studied the number of pores in samples obtained by this method depending on the percentage of 3D printing filling and direction of layers relative to the axis of the samples. The porosity of the samples was determined by comparing the theoretical ideal density with the experimental value determined using the hydrostatic weighing method. As a result, it was found that the porosity of the samples lays in the range from 17 to 18.5%. It was also found that with increasing percentage of filling the porosity of the samples increases. It has been suggested that this dependence is associated with a complication of the processes of air release from samples during the impregnation.

**Introduction**

In recent years, 3D printing technology is gaining more and more popularity. This fact is related to a number of advantages in comparison with traditional technologies, such as the ability of creating complex shaped products without special auxiliary tools (injection molds or press forms), in many cases lower cost of equipment, high speed of manufacturing parts due to the lack of a large number of technological operations, as well as the absence of the need for readjustment of equipment when changing the nomenclature [1]–[5]. One of the most common 3D printing technologies today is the FDM technology (fused deposition modeling, Figure 1). The stages of work look like this: polymer thread (filament) comes into the extruder where it melts; extruder moves along a given directory, forming a part layer; the layer is cured by cooling; cured layer shifts down; the extruder gives shape to a new layer, fusing it on top of the previous one, etc.

Despite the obvious advantages, 3D printing currently finds its main application for creating dummies that do not carry loads during operation. The creation of machine parts, which are able to work in real constructions under loads, is difficult due to the low mechanical properties of materials used for 3D printing. The strength of structures seeks to increase by printing continuous fiber reinforced composites [6], [7], as well as the creation of superengineering plastics for 3D printing.
However, these solutions require the use of expensive equipment for the printing process. The cost of the materials themselves is also very high.

**Figure 1.** Scheme of 3D printing using FDM technology: 1 — desktop; 2 — polymer thread (filament); 3 — feed rollers; 4 — heating element; 5 — product

One of the possible ways for increasing the strength properties of 3D-printed parts is its impregnation in a vacuum chamber in the thermosetting resins (Figure 2). The method is related to the vacuum infusion technology used to create woven composite [16]–[20]. Thermosetting resins have high strength properties compared to standard general-purpose plastics that are widely used for 3D printing. The main problem inherent in all similar methods of creating composites by impregnation is the residual porosity, which impairs mechanical properties. This work is devoted to the study of the influence of various 3D printing parameters of construction for the porosity of composite material, which is obtained in the process of impregnation.

**Figure 2.** Vacuum system for impregnation: 1 — chamber; 2 — a transparent cover; 3 — manometer; 4 — polymer compound; 5 — impregnated part; 6 — exhaust valve; 7 — shutoff valve; 8 — vacuum pump

**Materials and methods**

In this work, porosity was studied on impregnated samples intended for further tensile and toughness tests (Figure 3, a). Samples were printed on a Picasso 3D Designer XPro 3D printer using FDM technology with different percentages (20, 33 and 50%, Figure 3, b), as well as with different lay directions — along and across the axis of the samples (Figure 3, c). Printing material is a polylactide (PLA). The impregnation was carried out using the vacuum system shown in Figure 2. As an impregnation material was used epoxy diane resin with 20% of epoxy groups called ED-20 with a
polyethylene polyamine as a curative. Exposure in vacuum was 30 minutes. After the impregnation, the samples were cured at a room temperature for 24 hours.

**Figure 3.** Parameters of the testing samples: a — shape (on the left are standard samples for tensile tests, on the right — for impact tests); b — percentage of filling; c — print direction

The porosity of the samples was estimated by comparing the theoretical ideal density of the samples $\rho_t$ with the real determined by hydrostatic weighing $\rho_r$:

$$ P = \left( \frac{\rho_t - \rho_r}{\rho_t} \right) \times 100\% $$

The theoretical density of the samples was determined as follows:

$$ \rho_t = \rho_{PLA} \cdot V_{PLA} + \rho_{ED-20} \cdot V_{ED-20} $$

where $\rho_{PLA} = 1.24 \text{ g/cm}^3$ — printing material density; $\rho_{ED-20} = 1.21 \text{ g/cm}^3$ — cured compound density; $V_{PLA}, V_{ED-20}$ — volume fractions of material parts, $V_{PLA} + V_{ED-20} = 1$. The amount of plastic $V_{PLA}$ used for printing for each filling was determined using a Polygon X 3D printing slicer. The volume of epoxy resin for each samples filling was calculated as the difference between the volume of plastic required to print a sample with 100% filling and the volume of plastic required to print a frame of a certain filling.

The real density of the samples was estimated by hydrostatic weighing according to GOST 15139–69. The essence of the method is to compare the mass of the sample, measured in air, with the mass of the sample, measured by immersion in a working fluid of known density, in this study distilled water. Weight is determined using accurate scales with an error of not more than 0.001 g. The density of the sample is determined as follows:

$$ \rho_r = \frac{m_a}{m_w} \rho_w $$

where $m_a$ — sample weight in the air; $m_w$ — sample weight in the working fluid; $\rho_w = 1 \text{ g/cm}^3$ — density of the working fluid (distilled water).
Results and discussion
The results of the study of the porosity of the samples are presented in Figure 4. At each point, the average value of porosity calculated for a group of three samples is given. The confidence interval according to the calculation results ranged from 0.29 to 0.34. The porosity in the samples intended for tensile testing varies in the range from 17.5 to 18.5%, in the samples intended for impact testing — from 17.1 to 18.5%.

![Graph showing porosity vs. infill percentage for different sample types.](image)

Figure 4. Porosity of the tested samples: a — samples for tensile testing; b — samples for impact testing.

It can be seen that in all cases the value of porosity increases with an increase in the percentage of filling of samples in 3D printing. Moreover, the porosity of the samples intended for tensile testing has very similar values regardless of the orientation of the layers relative to the axis of the sample, while the porosity of the samples for impact tests differs: for samples of the “across” type it turned out to be slightly higher than for samples of the “along” type. In general, the porosity of the tensile test specimens is slightly higher than the porosity of the impact test specimens.

An increase in porosity with an increase in the percentage of filling can be explained by the fact that in the presence of a denser mesh inside the sample it is more difficult for air to go outside during vacuum impregnation. A slightly higher level of porosity in the samples for tensile tests compared with samples for impact tests can be explained by the fact that the former have a slightly larger volume compared to the latter, which means that the likelihood of an air bubble is higher.

Conclusion
As a result of the studies, the dependence of the porosity of the samples of composites based on 3D-printed frames impregnated with epoxy resin on the percentage of 3D printing filling was revealed. The higher the sample filling percentage, the higher the porosity was observed, which is probably can be explained by the fact that with a denser internal structure, it is more difficult for air to leave the sample during the impregnation process. On average, the porosity was about 18% and had a small variation depending on the parameters of the manufacture of the sample (within ± 0.5%). This level of porosity is large enough and can significantly affect the mechanical properties of the samples during testing, and therefore it is recommended to adjust the impregnation process to improve quality.

Acknowledgement
The reported study was funded by RFBR, project number 19-38-90037.
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