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Challenges in Tensile Testing of Thermoplastic Composites Reinforced with Chopped Carbon Fibre Produced by Fused Filament Fabrication Method

Jaroslav Majko (0000-0001-7136-2656), Milan Vaško (0000-0003-3585-2572), Marián Handrik (0000-0002-8622-3103), Milan Sága (0000-0002-0938-8772)
Faculty of Mechanical Engineering, University of Žilina. Univerzitná 8215/1, 010 26 Žilina. Slovak Republic. E-mail: jaroslav.majko@fstroj.uniza.sk

Additive manufacturing is a relatively new technology that has recently undergone noticeable development, which includes several types of technologies based on the gradual deposition of material in layers. The most widespread method is Fused Filament Fabrication, which belongs to an extrusion technique. The typical feature of extrusion methods is material deposition in the filaments form. Therefore, printer users cannot apply the same approach to products as with conventional technologies. The authors of the paper have been working with the mentioned technology for several years. The primary goal of the research is the investigation how printing parameters affect the mechanical properties of laminates reinforced with chopped carbon fibres. Based on experience and knowledge, the authors report in this article the most common challenges encountered in the preparation process of specimens for tensile testing. This knowledge can also help ordinary users of 3D printers, who also face these challenges without being aware of the impact of these pitfalls on mechanical properties.

Keywords: Tensile testing, Additive manufacturing, Fused Filament Fabrication, Chopped carbon fiber

1 Introduction

Industrial production in recent years has undergone a fundamental development due to the advancements characteristic of the increasing implementation of automation in production, the introduction of interconnectivity and the application of new materials [1]. The role of materials is crucial in development process, as they are one of the factors that reduce product weight while improving their properties. In the past, the fundamental breakthrough was the discovery of plastics, which currently make up a significant market share. However, in general, it is not suitable to use plastics in all areas of practice. Sometimes practice requires material properties that can be difficult to achieve. Therefore, materials composed of multiple components, such as walls made of stems and mud, have been used in ancient times. Currently, we refer to materials as composites, which according to the definition, are composed of two or more components on a macroscopic level, with resulting properties surpassing the properties of the components [2-3]. Generally, the composites consist of two fundamental parts - the matrix and the reinforcement. The role of the matrix is the maintenance of the object shape, protection of the reinforcement from the environment, and transfer of the loading to the reinforcement. The primary purpose of reinforcement is to strengthen the manufactured object [4]. The matrix and reinforcement functions can be fulfilled by various material types - metals, plastics, ceramics and so on. Regarding the shape, the reinforcement is divided into the following groups:

- Particles (Fig. 1a),
- Short fibers (Fig. 1b),
- Continuous fibers (Fig. 1c),
- Plates (Fig. 1d).

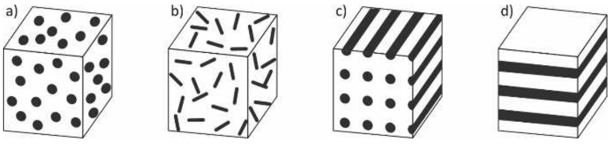


Fig. 1 *Various types of reinforcement: a) particles, b) short fibers, c) continuous fibers, d) plates*

Individual types of reinforcements have a significant impact on the composite properties. In addition, it is necessary to consider the material from which the reinforcement is composed and its arrangement in the structure [5-6]. Some of the main advantages of composites include a high weight-to-strength ratio, corrosion resistance, tailored properties, and design flexibility [7]. In certain circumstances, they also provide more cost-effectiveness than traditional materials, and their recyclability reduces the amount of waste and environmental impact. The main disadvantages of composites are complex manufacturing, limited recyclability, higher costs, and vulnerability to impact loading [8]. Other disadvantages represent complex failure, the need for quality control during the production process, energy-intensive production affecting the environmental impact, and in some cases, health risks associated with the release of hazardous substances during production [9-11].

Until recently, composite production was possible using conventional technologies (resin transfer moulding, compression moulding, vacuum bagging), with limitations primarily in the requirement to produce large quantities of complexly shaped products [12-14]. In recent times, the rapid development of additive manufacturing has led to innovations in many methods and technologies, which allow the printing of short-fibre reinforced composites. These methods are:

- Fused Filament Fabrication [15-18]
- Selective Laser Sintering [19-22]
- Composite-Based Additive Manufacturing [23]
- Stereolitography [24-25]

The mentioned methods differ significantly in processing procedure, with a significant impact on the properties of the printed object. Therefore, in this article, the working principle of the FFF method that the authors of the article work with will be described in more detail in the following chapter. Subsequently, the opportunities and challenges in the tensile testing of the thermoplastic composites reinforced with chopped carbon fibre that the authors of this article have encountered in practice will be described.

2 Fused Filament Fabrication

Fused Filament Fabrication (FFF) method belongs to the group of extrusion-based 3D printing methods. It is the most widely-used additive manufacturing technology in practice, whose usage was until recently limited only to the printing of thermoplastic materials [26-27]. However, in recent years, the rapid development of 3D printing has allowed the expansion of the range of materials that can be processed [28]. In the case of composite printing using FFF, no fundamental modifications to the printer equipment or its process are required. Nylon containing chopped carbon fibres is wound on a spool in the form of a filament and gradually fed into the printer head, which is heated to the materials' melting temperature. The heated material located in the nozzle is then dropped on the printing desk and bonded with the previously deposited filaments. When the printer finishes the lamina printing, the printing desk drops by the thickness of the given lamina and continues printing the next layer until the entire object is complete (Fig. 2).

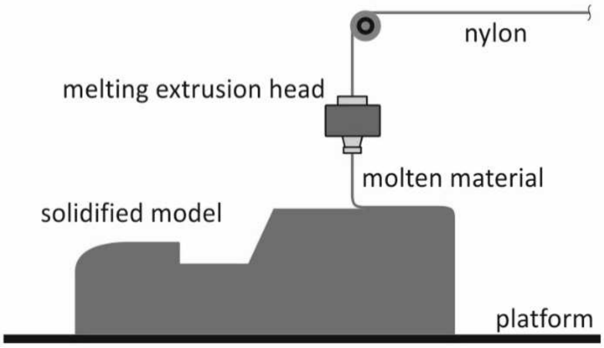


Fig. 2 *Scheme of printer working on FFF principle*

The advantages of the FFF technology are low investment and operating costs, a wide range of usable materials, ease of working with them, and relatively high printing accuracy and reliability [29-31]. The disadvantages of the method are warping caused by cooling of the deposited material, delamination, lower strength of objects compared to conventional technologies, and stringing [32].

3 Tensile testing

Tensile tests belong to a class of static testings, which principle is based on slow controlled tensile loading of the test specimens of prescribed shapes and dimensions until failure occurs. The method of clamping the specimen into the jaws of the testing machine must be such that the axis of the specimen coincides with the axis of the applied load. During the test, a force-displacement diagram is recorded, showing the dependence of the specimens' deformation on the applied loading (Fig. 3).

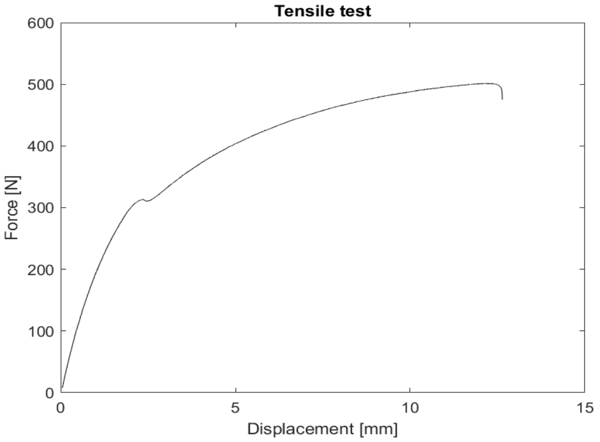


Fig. 3 *Force - displacement diagram*

In the vast majority of articles published to date focused on tensile testing of additively produced fibre-reinforced composites, the authors have applied the ASTM D638-14 standard [33], originally designed for plastics produced using conventional technologies. The characteristic of this standard is the dog bone-shaped specimen, meaning a narrowed area in the centre of the specimen.

4 Challenges

As additive manufacturing significantly differs from conventional technologies based on material removal, the following paragraphs will describe certain limitations that a user of a 3D printer based on extrusion methods must consider in practice (especially under tensile loading).

4.1 Specimen shape

As mentioned in Chapter 3, in the case of additively manufactured composites tensile testing predominates dog bone-shaped specimens (Fig. 4).

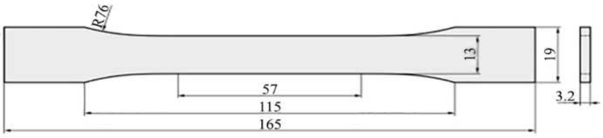
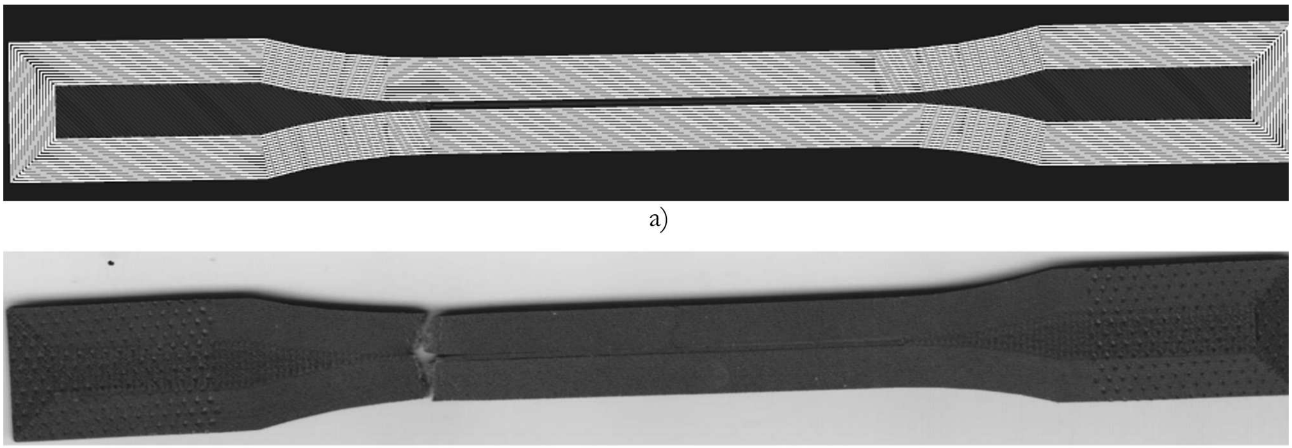


Fig. 4 *Specimen shape according to standard ASTM D638-14*

The shape is appropriate for homogeneous materials since failure generally occurs in the narrowest part of the specimen. The reason is the highest stress in this location, which exceeds the strength of the material and causes specimen breakage. In the case of inhomogeneous materials produced by the extrusion method, a similar approach to tensile testing is relatively problematic due to the deposition of the material in the form of filaments (Fig. 5). Experience shows that under certain circumstances, failure often occurs in the area of the rounding [34]. The cause is the presence of a stress concentrator. It means that the strength of the material in that configuration is higher than the result obtained from the tensile test.

Another significant disadvantage of this type of specimen failure is the presence of cracks out of the gauge length, which creates difficulties in displacement measurements (in both the axial and transverse directions). In addition, the strain is defined as the ratio of displacement to the total length in the axial direction, which in this case, due to the varying orientation of the filament in the roundings area, does not correspond to reality. According to the authors' experience, filament deposition around specimen circumference (in 3D printing terminology, "walls") leads to failure at inappropriate locations. Strategies without walls usually crack in the narrowed area (Fig. 6). However, the deposition around the specimen circumference has a strengthening effect and, despite the occurrence of stress concentrators, leads to the highest measured tensile strength values [34].



a)

b)

Fig. 5 *Specimen with filament arrangement around the circumference: a) screenshot of the slicing software, b) photo of the specimen with a crack in an inappropriate area*

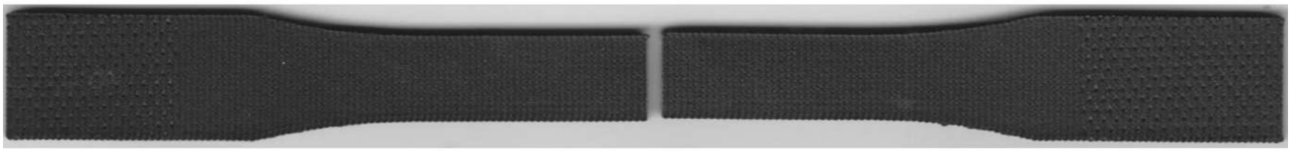


Fig. 6 *Failure of the specimen without walls; The crack is located in the gage length area*

4.2 Location of filament end

A crucial requirement in the design process of specimens is considering the fibre end location. The inappropriate fibre end location can lead to premature failure outside of the gauge length area due to high shear stresses [35] and the presence of stress concentrators.

Fig.7 shows the fibre end location at the end of the rounding (highlighted by the red circle), which will lead to specimen failure outside the narrowed area. In addition, the specimen gripping by the jaws of the machine [36] can also affect specimen behaviour.

Fig. 7 *Inappropriate placement of the filament leads to premature failure of the specimen.*

4.3 Filament orientation in the structure

During the printing preparation process, the user must take into account the filament orientation in the structure (Fig. 8). There are many material arrangement strategies - for example, hexagonal infill, triangular infill, 45/-45, 0/90 [34, 37-38]. Most of them must preset the user manually in the slicing software (infill pattern). However, others can be modified without adequate knowledge of slicing software, for instance, by changing the object orientation on the printing bed because the dimensions of the printed object exceed the printing bed dimensions.

Generally, the highest strength achieves specimens with filament oriented axially to the load axis. Interesting results also have specimens with arrangement 45/-45, which increases the ductility several times. In the case of the 0/90 strategy, the disadvantage is that approximately half of the laminates are oriented perpendicular to the load axis, which means a significant reduction of strength due to the presence of bonds between adjacent filaments, which are relatively weak in the case of extrusion methods of additive technologies. The results show a considerable directional dependence on mechanical properties, whereas objects printed using this technology behave orthotropically [38-40]. Typically, the worst tensile properties are obtained in the layering direction of the laminates [40-41].

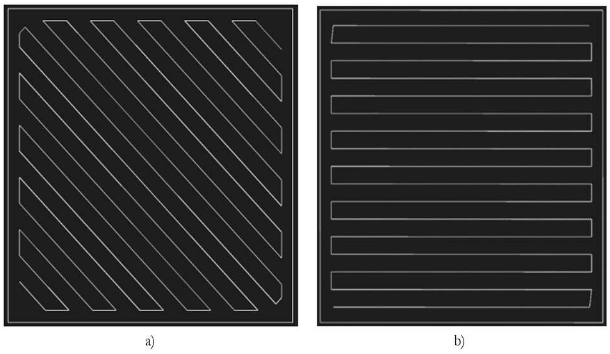


Fig. 8 *Filament orientation in the lamina: a) 45 degrees, b) 0 degrees*

4.4 Voids in the structure

One of the key drawbacks of extrusion techniques is the technology's imperfection accompanied by a high incidence of voids in the structure. The reason is the deposition of material from the nozzle onto the printing bed [42], low filament feed rate [43], and layer thickness [44]. The orientation of the filaments in the structure significantly affects the occurrence of voids between filaments, with a higher proportion of defects observed in the 0/90 strategy compared to the 45/-45 strategy (Fig. 9).

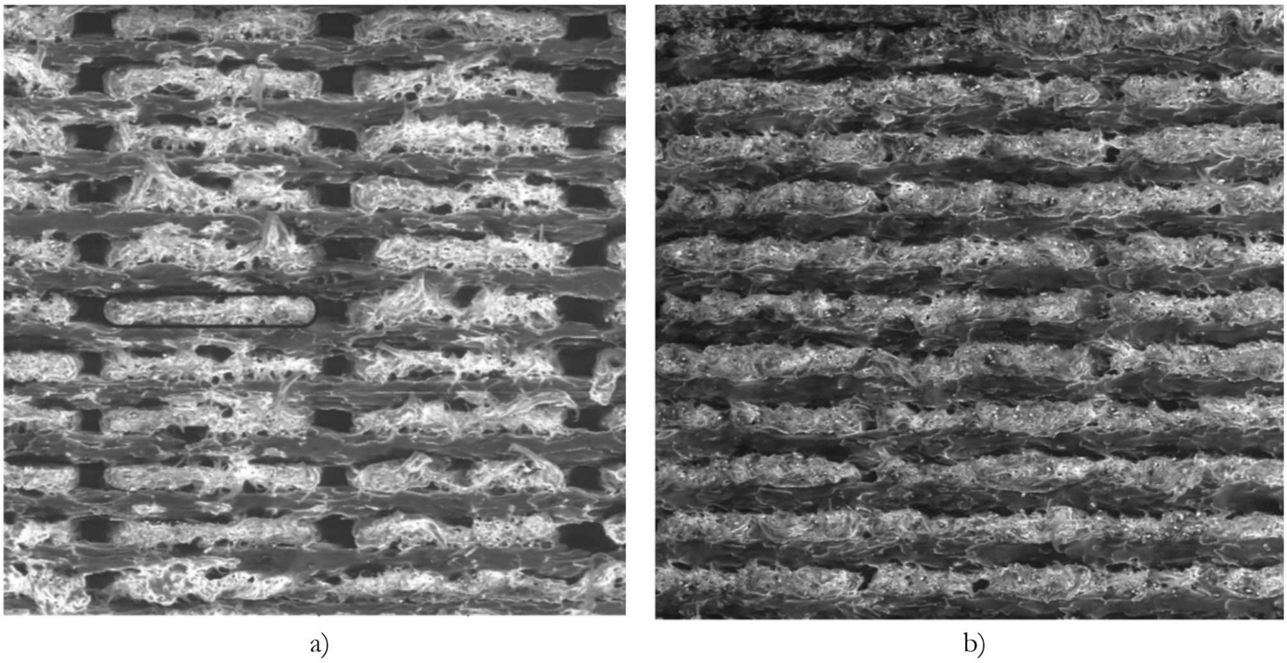


Fig. 9 *Image of voids in the structure printed by extrusion method: a) filament orientation in 0/90 layers, b) filament orientation in 45/-45 layers*

The presence of voids in the structure also affects the formation of stress concentrators, which results in a weakened structure of printed objects. In the case of methods (such as sintering), the density of printed objects is high, resulting in substantially higher strength of printed objects compared to processing by conventional technologies [45].

4.5 Moisture

Nylon is a hygroscopic material [46], for that reason, proper storage of this material before the printing process is necessary since the moisture absorption leads to a decrease in the melting temperature of nylon [47]. If the printer user does not adjust or cannot adjust the temperature of the nozzle, the nozzle dripping will occur. One of the outcomes is uncontrollable deposition on the printing desk. The result are strings (Fig. 10), voids, and imperfections, which negatively affect aesthetic appearance and also the mechanical properties (strength, strain, failure type) of printed objects. For this reason, the printing of wet material is problematic, even unreal, and therefore is highly recommended to store nylon in suitable conditions in closed containers.

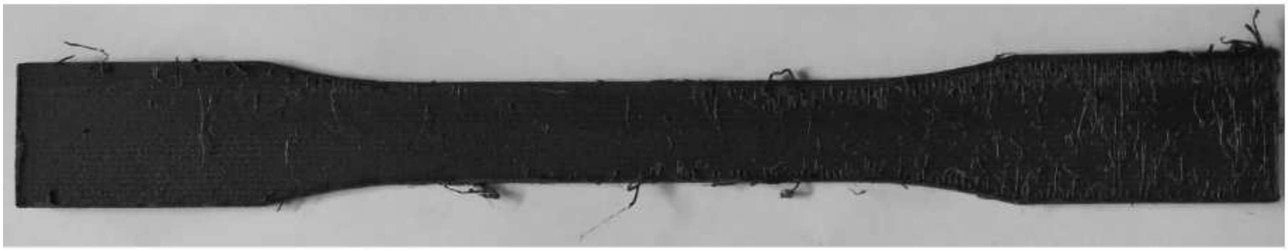


Fig. 10 *Consequence of inappropriate material storage*

According to our experience, one of the possibilities to print wet nylon is the modification (decrease) of the nozzle temperature. However, the result is weak interlaminar bonding that can be eliminated by increasing the temperature of the printing bed. The results are comparable to the strength of specimens from appropriately stored material [48]. The used material was exposed to room temperature and humidity for a long time before the printing process. The measurement of the specimens' humidity was not performed. At the same time, the printer user must take into account that the material after printing can absorb moisture, which also affects mechanical properties [49]. The authors prepare a comprehensive study, which will assess various humidity conditions influence before and after printing on the tensile properties of specimens.

4.6 Layering

The working principle of additive manufacturing is based on the gradual layering of material [50]. Each layer has a thickness of approximately 0.1 or 0.2 millimetres. According to the ASTM D638-14 standard, the thickness of the specimens is 3.2 millimetres with a tolerance of ±0.2 millimetres [33]. The reason is that the sufficient number of layers ensures a representative interpretation of the results. If the specimen consists of a low number of layers, it would not be possible to consider it macroscopically homogeneous, as different and even excessive numbers of defects in the structure could occur, resulting in significant differences in the measured tensile properties of the printed specimens. Based on the data in Table 1, the authors observed a substantial decrease in the values of standard deviations with an increasing number of layers. The values of average engineering yield stress were calculated from average yield forces divided by original crossectional area.

Tab. 1 *The increasing number of layers leads to a reduction of the standard deviation*

No. of Laminas	Average yield force [N]	Average yield stress [MPa]	Standard deviation [MPa]
5	187.47	23.07	0.9641
7	251.24	22.09	0.392
9	339.22	23.19	0.2595

5 Conclusion

The authors of this article aimed to present fundamental problems encountered during the realization of tensile tests conducted on nylon reinforced with chopped carbon fibre. The results are the principles appropriate for preparing the experimental measurement that should be the basis for the implementation and solid evaluation of the measured outcomes. All mentioned parameters can significantly affect the reliability of this material in practice.

From the author's experience in the preparation process of the tensile test, it is crucial to consider the suitability of applied parameters. The first parameter is specimen shape since the shapes defined in currently valid standards are proposed solely for conventional technologies. In the case of material extrusion-based additive manufacturing methods are materials gradually deposited in the form of filaments. Therefore, the shapes defined in these standards may not be perfectly correct for tensile properties evaluation. For example, filaments deposition around the circumference of the specimen leads to failure out of the gage length area. However, other strategies without walls usually lead to a rupture in the gauge region. At the same time, concerning the occurrence of stress concentrators, the user must necessarily consider the localization of fibre ends. In addition, the infill orientation of filaments concerning the load axis is a printing parameter that substantially affects tensile properties. The assessment of the fracture surfaces also identifies the effect of filament arrangement on the presence of voids in the structure. Hence the primary aim of the current research is the reduction of voids, as they negatively affect the strength of printed products. In order to suppress the randomness of printing defects, the user must choose a sufficient number of layers of the laminate. For that reason, the standard requires 3.2 millimetres thick specimens. The authors compared the yield strength of specimens with the different numbers of layers (5, 7, and 9). The assessment of results identified a decreasing spread of the measured results with the increasing number of laminas. An inappropriately stored nylon absorbs moisture, which highly affects the printing process. By default, material overflows from the nozzle during printing, resulting in poor print quality and a significant decline in mechanical properties. Additionally, nylon absorbs moisture after the printing process.

All of these factors have a significant impact on the measured tensile properties of thermoplastic composites reinforced with chopped carbon fibres. 3D printer users must deal with these parameters to avoid potential problems with the application of printed objects in practice, which can lead to unpleasant and unexpected consequences during operation.

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